This is a digital copy of a book that was preserved for generations on library shelves before it was carefully scanned by Google as part of a project to make the world's books discoverable online.

It has survived long enough for the copyright to expire and the book to enter the public domain. A public domain book is one that was never subject to copyright or whose legal copyright term has expired. Whether a book is in the public domain may vary country to country. Public domain books are our gateways to the past, representing a wealth of history, culture and knowledge that's often difficult to discover.

Marks, notations and other marginalia present in the original volume will appear in this file - a reminder of this book's long journey from the publisher to a library and finally to you.

Usage guidelines

Google is proud to partner with libraries to digitize public domain materials and make them widely accessible. Public domain books belong to the public and we are merely their custodians. Nevertheless, this work is expensive, so in order to keep providing this resource, we have taken steps to prevent abuse by commercial parties, including placing technical restrictions on automated querying.

We also ask that you:

+ *Make non-commercial use of the files* We designed Google Book Search for use by individuals, and we request that you use these files for personal, non-commercial purposes.

+ *Refrain from automated querying* Do not send automated queries of any sort to Google's system: If you are conducting research on machine translation, optical character recognition or other areas where access to a large amount of text is helpful, please contact us. We encourage the use of public domain materials for these purposes and may be able to help.

+ *Maintain attribution* The Google "watermark" you see on each file is essential for informing people about this project and helping them find additional materials through Google Book Search. Please do not remove it.

+ *Keep it legal* Whatever your use, remember that you are responsible for ensuring that what you are doing is legal. Do not assume that just because we believe a book is in the public domain for users in the United States, that the work is also in the public domain for users in other countries. Whether a book is still in copyright varies from country to country, and we can't offer guidance on whether any specific use of any specific book is allowed. Please do not assume that a book's appearance in Google Book Search means it can be used in any manner anywhere in the world. Copyright infringement liability can be quite severe.

About Google Book Search

Google's mission is to organize the world's information and to make it universally accessible and useful. Google Book Search helps readers discover the world's books while helping authors and publishers reach new audiences. You can search through the full text of this book on the web at `http://books.google.com/`

KF
23254
NEDL TRANSFER
HN 4YFX /

METALLURGICAL LABORATORY NOTES

HENRY M. HOWE

HARVARD ENGINEERING SCHOOL
LIBRARY OF MINING AND METALLURGY

BOSTON TESTING LABORATORIES

KF23254

E11 30.5:10

HARVARD UNIVERSITY
LABORATORY OF
MINING AND METALLURGY

Given by
MISS SUSAN T. CUSHING

FROM THE LIBRARY OF
DR. EDWARD DYER PETERS
Professor of Metallurgy in Harvard University
1904–1917

ROTCH LIBRARY

METALLURGICAL LABORATORY NOTES

BY

HENRY M. HOWE

Professor of Metallurgy in Columbia University
in the City of New York

PUBLISHED BY THE
BOSTON TESTING LABORATORIES
Boston, Mass., U. S. A.
1902

30.5.10

TN
665
36

HARVARD UNIVERSITY
MINING AND METALLURGICAL
LABORATORY
JAN. 20, 1922
GIFT OF
MISS SUSAN T. CUSHING

KF23254

Copyright, 1902, by
ALBERT SAUVEUR

HARVARD
UNIVERSITY
LIBRARY
JUL 28 1954

Trans to HCL

Languages Printing Company, 114 Fifth Avenue, New York

To

THOMAS MESSENGER DROWN, M.D., LL.D.

FRIEND, CHEMIST, EDUCATOR

As a token of affectionate regard, this work is dedicated

PREFACE

METALLURGICAL laboratory instruction of the established type reproduces the industrial metallurgical processes, each as a whole, and with such fidelity to actual industrial practice as is attainable. This, which we may call "Applied Metallurgy," seems to me to have inherent and very grave faults. In its place I have tried to work out a system of what we may call "Theoretical" or better "Analytical Metallurgy," with the aim of teaching, not individual processes each as a whole, but the individual underlying principles, each by itself. This I have tried to do by means of a series of distinct experiments, each testing directly one or more of these principles.

This little book represents the results of my attempt, which, if we leave out of consideration works on the kindred but really distinct subject of assaying, is, so far as I know, the first serious and systematic attempt in this direction. May it receive the indulgence to which such first attempts are entitled. Its limitations, especially its lack of balance, its devotion chiefly to certain branches of metallurgy, I feel only too keenly. But I have been confronted not only with a theory but with a condition, that of preparing, with very limited time and energy, some kind of a system for students present, commanding attention, and unable to await the completion of a well rounded work. I have therefore done what I could rather than what I would, preparing experiments upon those branches of metallurgy with which I happen to be familiar.

I have endeavored to smooth away every obstacle, with the object not of saving the student trouble, but of enabling him to go farther with a given expenditure of energy, and of permitting me to divert his attention from the laborious details of manipulations to the unwelcome task of thinking. By means of extremely

elaborate directions I try to avoid the waste of the student's time which the task of discovering for himself suitable conditions would cause. I try to concentrate his attention in each experiment upon the special principle which that experiment illustrates. I require that he shall formulate in terms his inferences from his experiments. Thus my aim is to compel the greatest amount of thinking and of careful and exact observation, and to reduce to a minimum the play or mechanical side of laboratory practice, its reasonable reproach.

A word as to the relative merits of the applied and analytical systems of teaching metallurgy in the laboratory may not be amiss here.

If the aim of our metallurgical laboratory study is to anticipate industrial practice, we should follow the applied metallurgy plan: if its aim is to teach principles rather than practice, we should follow the analytical plan. It seems clear on general grounds that instruction in general should so far as possible deal with principles rather than with the details of practice. But additional reasons, if any are needed, exist in case of metallurgical laboratory instruction. For the details of practice are learnt inevitably, spontaneously, accurately, and with the greatest ease in practice itself, but with very great difficulty and distortion in the laboratory. Principles, on the other hand, are what the laboratory teaches most readily and perfectly, and practice least readily and most imperfectly. The path of least resistance then is to study principles in the laboratory and the details of practice in practice itself.

The divergence between the applied and analytical systems of teaching may be illustrated by the selection of furnaces. I have elsewhere* said "his furnaces should in general be heated "by gas or electric resistance, so that his attention may be con- "centrated on the phenomenon which he is studying, and not "diverted to keeping a coal fire in proper condition." To this an eminent educator, who follows the applied metallurgy type of instruction, replies:

"I think it very important that a little coke work should be "given for this reason: with your gas furnace you can regulate "and get your exact temperature and have it stand hour after

* *Science,* N. S., Vol. XV, p. 761, May 16, 1902.

" hour with very narrow margin of variation. That gives you " your mathematically correct, or nearly so, idea. Now, that is " not what the great works do. They have coke or coal which " has to be put on and fired up and then the drop comes when " more coke is added, and it is a very useful thing for our students " to realize this variation which comes with coke or solid fuel " and which does not come with gas as a fuel."

I answer that I indeed provide a single experiment, number 20, to illustrate this point. But I would no more hamper the whole of my student's course by the continued use of these inefficient coal furnaces throughout his other experiments, merely to impress farther on him the difference between the accuracy needed in investigation and the roughness appropriate to the manufacturing operations themselves, than I would force my students to use bad balances and transits throughout their course in order to impress on their minds the shortcomings to which those instruments are subject, and their remedies.

The truth is that the metallurgist has to practice both as a manufacturer, and as an investigator of the conditions of his manufacture and of the qualities of his product. The two functions are in a great measure distinct, like walking and swimming. In the manufacture proper great accuracy is rarely appropriate; in investigating the processes and products of the manufacture the greatest accuracy may be needed. Now, just as the water is the place to teach swimming and the land to teach walking, so the industrial establishment is the place to teach the degree of accuracy appropriate for manufacture, and the laboratory to teach the greater accuracy which these investigations will need; more especially because this kind of accuracy the industrial establishment does not teach.

While I hold these views, yet, as the art itself of teaching must be chiefly empirical, and as changes should therefore be made tentatively and their merit tested, I have elaborated also experiments for teaching certain industrial processes in the older or applied metallurgy method, so that the working of the two methods may be compared.

" Another and important work of the metallurgical laboratory " is to give a certain skill in the use of the instruments of pre- " cision of the art, in pyrometry, calorimetry and the microscopy " of metals and alloys. It seems to me nearly as imperative that

" the metallurgist's diploma to-day should imply this skill as that " the civil engineer's should imply skill in the use of the transit." *

With a very few exceptions all the experiments in this work have been carried out either in the metallurgical laboratory of the School of Mines of Columbia University, or earlier in the author's private laboratory, and either exactly in the form here given, or in a form of which the present is a slight modification, and it is hoped an improvement.

While most of these experiments are brief, yet there are some of which each is sufficiently extended to serve as an investigation for a graduation thesis, and others of intermediate length.

This work would have been impossible but for the zealous coöperation of the officers and students of the School of Mines, on whom the labor of the actual manipulations has fallen. My heartiest thanks are due to them, and especially to Messrs. Bradley Stoughton and E. J. Hall, whose devotion to the work has known no bounds.

The gas furnaces used, in addition to the well known Fletcher and Forquignon furnaces, are those of the American Gas Furnace Company. To its President, Mr. E. J. Reichelm, thanks are due for the great pains which he has taken in designing special furnaces for this work.

The experiments of Group I are designed especially to enable a few instructors to give to a large class of students a slight smattering of metallurgical conditions. To this end it is arranged that the students work in squads, each under a captain chosen from among them by the instructors; and the directions are given in very great detail.

The tables at the end of the work are original here, except that of atomic weights, and also except in a sense that of the melting points of silicates.

H. M. H.

COLUMBIA UNIVERSITY IN THE CITY OF NEW YORK,
June, 1902.

---

* Loc. cit.

TABLE OF CONTENTS

GROUP 1. — SQUAD EXPERIMENTS AND OTHERS FOR A SHORT COURSE IN METALLURGY

EXPERIMENT		PAGE
1.	Le Chatelier Thermo-Electric Pyrometer	3
2.	Temperature Limits of Hardening Power of Steel	10
3.	Metcalf's Experiment on Heat-Treatment of Steel	12
4.	"Refining" Restores Ductility of Steel	14
5.	Microscopic Metallography, Steel and Copper-Silver Alloys	17
6.	Oxidizing Roasting of Copper Sulphide Ore	19
7.	Parke's Process for Desilverizing Lead	24
8.	Tempering Steel	27
9.	Removal of Effects of Overstrain in Iron by Reheating	29
10.	The Hardening of Steel is a Function of the Carbon-Content	29

GROUP 2. — PYROMETRY AND CALORIMETRY

11.	Siemens' Water Pyrometer, Method of Mixtures	30
12.	Berthelot Calorimeter	31
13.	Le Chatelier Pyrometer. Melting Point of Copper	38
14.	Le Chatelier Pyrometer. Melting Point of Platinum	38
15.	Optical Pyrometers of Le Chatelier and Mesuré and Nouel	40
16.	Thermal Gradient	41
17.	Thermal Gradient	42
18.	Thermal Gradient	43
19.	Isotherms	43
20.	Relative Uniformity of Gas and Direct-fired Furnaces	45

GROUP 3. — MELTING POINTS OF SILICATES, ETC.

	General Directions Concerning the Determinations of Melting Points of Silicates and Other Experiments of Group 3	46
21.	Seger Pyramids	53
22.	The Influence of Fluorine on the Fusibility of Earthy Silicates (Singulo and Acid Silicates of Lime and Magnesia, $2CaO,MgO$)	53

EXPERIMENT		PAGE
23.	Melting Points of Lime-Magnesia Sesqui-Silicates	54
24.	Melting Points of Lime-Magnesia Tri-Silicates	54
25.	Melting Points of Lime-Magnesia 2.5 Silicates	54
26.	Melting Points of Lime Silicates	54
27.	Melting Points of Lime-Magnesia Silicates	55
28.	Melting Points of 2 CaO,MgO Silicates	55
29.	Melting Points of 3 CaO,MgO Silicates	55
30.	Melting Points of Lime-Alumina Silicates	55
31.	Formation Point and Melting Point of Silicates	56
32.	Verification of Relation of Formation Point to Melting Point	57
33.	Verification of Relation of Formation Point to Melting Point	58
34.	Verification of Relation of Formation Point to Melting Point	58
35.	Fusibility of Lead Silicates as affected by Acidity	59
36.	Fusibility of Lead Silicates as affected by Addition of Lime and Ferric Oxide	60

GROUP 4. — PROPERTIES OF REFRACTORY MATERIALS

37.	Contraction of Refractory Materials on Heating	60
38.	Contraction of Refractory Materials on Heating	61
39.	Slacking of Basic Refractory Materials	62
Corrosion of Divers Refractory Materials by Silicates. General Directions		62
40.	Action of Ferrous Singulo-Silicate ($2\,FeO,SiO_2$)	67
41.	Action of Ferrous Bi-Silicate (FeO,SiO_2)	67
42.	Action of an Earthy Singulo-Silicate	67
43.	Action of an Acid Earthy (2.5) Silicate	68
44.	Action of an Acid Silicate of Lime and Ferrous Oxide	68
45.	Action of a Basic Silicate of Lime and Ferrous Oxide	68

GROUP 5. — IRON AND STEEL

46.	The Desulphurization of Iron by Manganese	69
47.	Verification of Roberts-Austen's Diagram	72
48.	Manganese Steel	73
49.	Influence of Rate of Cooling on Fracture of Cast Iron	73
50 to 55.	Brinell's Fracture Experiments	75-78
56.	Growth of the Effects of Overstrain	78
57.	Refining Overheated Steel, Full Examination	80
58.	Burning Hypo-Eutectic Steel, Full Examination	82
59.	Refining Overheated Steel, Brief Examination	83

EXPERIMENT PAGE

60. Burning Hypo-Eutectic Steel, Brief Examination . . . 83
61. Influence of $T^{max.}$ on the Thoroughness of Refining, Full Examination 83
62. Influence of $T^{max.}$ on the Thoroughness of Refining, Brief Examination 84
Finishing Temperature. General Directions 84
63. Finishing Temperature for Rail Steel 87
64. Finishing Temperature for Structural and Boiler Plate Steel 88

MICROSTRUCTURE

65. Microstructure of Blister Steel 88
66. Distribution of Martensite 89
67. Distribution of Martensite 89
68. Metcalf's Experiment Elaborated 90
69. Metcalf's Experiment Elaborated 90

GROUP 6. — NON-FERROUS METALS

COPPER

70. Copper "Selecting" Process 91
71. "Spelter Test" for Purity of Copper 92
72. Copper Unites with Sulphur in Preference to Oxygen . . 93
Chloridizing Roasting. General Directions 94
73. To Find the Best Temperature 96
74. To Find the Best Percentage of Raw Pyrites . . . 97
75. To Find the Minimum Quantity of Salt 97
76. To Find the Most Economical Conditions 97
77. To Show the Influence of Sulphur 98
78. To Show the Influence of Ferric Oxide 98
Ziervogel Process. General Description 98
79. Ferrous Sulphate for Sulphatizing Copper . . . 101
80. Sulphatizing Action of Ferrous Sulphide 102
81. Silver Sulphide when Roasted Yields Metallic Silver and Silver Sulphate 102
82. The Dissociation of Cupric Sulphate Stimulates the Sulphatizing of Silver 102
83. The Spangle Reaction 103
84. Sulphatizing Metallic Silver by Ferrous Sulphate . . 103
85. Sulphatizing Metallic Silver by Cupric Sulphate . . 104
86. Copper Electrolysis 104
87. Alloy Diffusion Experiment of Le Chatelier and Stead . . 107

GOLD AND SILVER

EXPERIMENT		PAGE
88.	Cyanide Process	108
	Chlorination Process, General Directions.	
89.	Chlorinating a Normal Ore	113
90.	Chlorinating in the Presence of Raw Pyrites	113
91.	Chlorinating in the Presence of Ferrous Sulphate	114

APPENDIX

Directions for Lining Crucibles.

	Brasquing	115
	Material	115
	Ramming	115
	Basic and Neutral Linings	116

TABLES

NUMBER		
4.	Atomic Weights	117
5.	Molecular Weights, etc., of Various Inorganic Compounds and their Percentage Composition	118-121
6.	Synoptical Table for Metallography	122-123
7.	Melting and Boiling Points, etc.	124-125
8.	Melting Points of Silicates (Slags)	126
9.	Comparison of Thermometric Scales	127
10.	List of the Chief Special Apparatus Needed for the Experiments of this Book	128

LIST OF FIGURES

FIGURE		PAGE
1. 2.	Le Chatelier Thermo-Electric Pyrometer	4
3.	Apparatus for Determining Boiling Points of Water and Sulphur	6
4.	Apparatus for Determining Melting Point of Zinc	7
5.	Arrangement for Determining Critical Temperature of Steel	8
6. 6a. 6b.	Arrangement of Wire, etc., for Experiment 2	10
7.	Bar Nicked for Metcalf's Experiment	13
8.	Drop-Testing Machine	15
9.	Oven Furnace (American Gas Furnace Co.)	20
10.	Tensile Testing Machine for Experiment 8	28
11.	Mahler's Berthelot Bomb Calorimeter	32
12.	Cooling Correction for Calorimetry	35
13.	Electric-Resistance Crucible Furnace	37
14.	Arrangement for Calibration by Melting Point of Platinum	39
15.	Tubular Muffle	42
16.	Positions of Couple in Muffle for Thermal Gradient Experiments	44
17.	Mould for Silicates. Horizontal Section	46
18.	Mould for Silicates. Vertical Section	46
19.	Gas Forge Showing Muffle Arrangement for Silicate Melting Point Determinations. Cross-Section	48
20.	Gas Forge Showing Muffle Arrangement for Silicate Melting Point Determinations. Longitudinal Section	49
21.	Special Muffle for Silicates, etc. Cross-Sections	50
22.	Special Muffle for Silicates, etc. Longitudinal Section	51
23.	Chromite Insulating Wafer	51
24.	Arrangement of Crucibles for Experiments on Corrosion of Refractory Materials. Cross-Section	64
25.	Plan of Graphite Slab used for Supporting Crucibles	64
26.	Crucible Furnace (American Gas Furnace Co.)	66
27.	Roberts-Austen's Diagram	70-71
28.	Cast Iron Ingot Mould for Experiment 49	74
29.	Clay Ingot Mould for Experiment 49	74
30-35.	Graphical Representation of Brinell's Fracture Experiments	76
36.	Gas Forge Showing Arrangement of Bars for Experiment 57. Cross-Section	80

FIGURE		PAGE
37.	Gas Forge Showing Arrangement of Bars for Experiment 57. Longitudinal Section	81
38.	Arrangement of Bars in Detail	81
39.	Rolls for Finishing Temperature Experiments	85
40.	Electric-Resistance Tubular Muffle Furnace	86
41.	End of Electric-Resistance Furnace, Enlarged	87
42.	Tree Illustrating Ziervogel Process	99
43.	Copper Electrolysis	106
44.	Chlorinating Barrel	110

METALLURGICAL LABORATORY NOTES

GROUP 1.—SQUAD EXPERIMENTS AND OTHERS FOR A SHORT COURSE IN METALLURGY

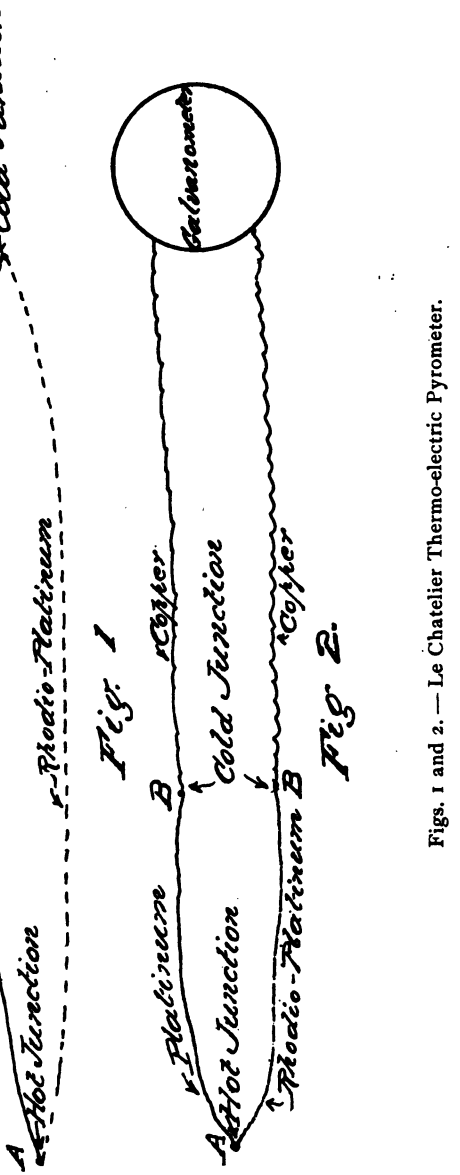
Preliminary Note

THE experiments performed by squads in general occupy two afternoons. Each student will be assigned his work by squad and number, and in order that it may be as varied as possible the numbers will be different for the first and second afternoons in each subject. No experiment is to be considered complete until its results have been approved and initialed by the designated officer. Students leaving the laboratories before the results have been thus initialed will be counted as absent.

Experiment 1

THE LE CHATELIER THERMO-ELECTRIC PYROMETER. — *Principle of the Instrument.* — If wires of two different metals, in this case (1) pure platinum and (2) platinum alloyed with 10 per cent of rhodium, be joined at both ends, and if one junction (A, Fig. 1) then be heated and the other junction kept cool, a slight electric current flows through them because of the difference between the temperature of their two junctions, increasing in strength as that difference increases. The same is true if, as sketched in Fig. 2, their cool ends, instead of being joined directly, are joined by means of an intermediate wire of copper, in the course of which lies a galvanometer. As before, if the junction A be heated hotter than the junction B, a current flows through the wire because of that difference of temperature; and by measuring with the galvanometer the strength of that current, we can calculate the difference of temperature between the hot and the cold junction. In order to do this we first "calibrate" the instrument by observing how great a deflection of the galvanometer is caused by exposing the hot junction A to a series of known temperatures readily reproduced, such as the boiling points of water, sulphur, and zinc, and the

melting points of zinc, aluminium, copper, and platinum, keeping the other or " cold " junction B at known low temperature. Having thus calibrated the instrument, we can ascertain the tempera-



Figs. 1 and 2. — Le Chatelier Thermo-electric Pyrometer.

ture of any part of a furnace, a crucible, or other hot body, by setting in it the junction A, keeping the junction B at a known low temperature, and noting the strength of the current produced.

Subdivision of Squad. — The squad will divide into two sections. Section 1, including the first Captain and Nos. 2 and 3, will work with the pyrometer on the south side of the furnace room; section 2, including the second Captain and Nos. 4 and 5, will work with the pyrometer on the west side of the furnace room. Each section will work wholly independently of the other.

Preliminary Practice in Reading. — Insert the thermo-junction in fine clay in a clay crucible about one inch in diameter, in a Forquignon or Fletcher crucible furnace. Take care that the leads do not short-circuit. Light the furnace and record on one of the blanks provided the times at which the cross hair or the needle of the galvanometer passes each division of the scale as the temperature rises. After reaching a light yellow heat cut off the gas, and in the same way record time and temperature during cooling. Repeat this.

Test the accuracy of the work by comparing the intervals of time between the successive divisions of the scale. With a little practice they will be very regular.

Record the zero every fifteen minutes, and the temperature of the cold junction at least every hour. Do this in all subsequent work with the pyrometer.

General Plan of Work. — Calibrate the instrument by noting the deflections of the galvanometer caused by exposing the thermo-electric junction to boiling water, 100° C., boiling sulphur, 445° C., and melting zinc, 420° C. Plot with temperature as ordinate and deflection as abscissa, a curve passing through the points thus determined. This curve is to be used for translating the deflections of the galvanometer into degrees Centigrade in the subsequent work, e. g., the determination of the critical temperature of steel of 0.80 per cent of carbon.

Details of Calibration. Precautions. Cold Junction. — The electric current is due to the difference between the temperatures of the two junctions; the deflection of the galvanometer is therefore determined, not simply by the temperature of the thermo-junction A, but by the difference between its temperature and that of the cold junction B, Fig. 2. Therefore this cold junction must be immersed in a large vessel of water at known tem-

perature. For every reading the corrected temperature of the thermo-junction is that indicated by the deflection of the galvanometer, plus that of the cold junction.

If the leads are springy or kinked, anneal them by heating to redness from one end to the other in a Bunsen burner.

Short-Circuiting. — Should the leads cross anywhere between the thermo-junction and the galvanometer, this will short-circuit the current, which therefore will not deflect the galvanometer.

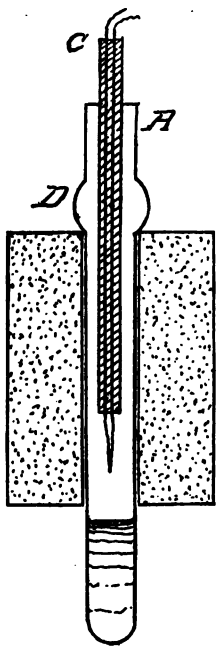


Fig. 3. — Apparatus for Determining the Boiling Points of Water and Sulphur.
Scale 1 = 2.

Calibration in Boiling Water and Sulphur. — Fill a test-tube A, Fig. 3, with water or sulphur as shown, surrounding the test-tube with a plaster-of-Paris block to insulate the heat. Insert the thermo-junction as shown, the wires being prevented from short-circuiting by the double-bored clay tube C through which they pass. Heat the water or sulphur until the test-tube is filled with steam, or till sulphur condenses abundantly in the

swelling D. Then insert the thermo-junction into the water itself, or into the fumes of sulphur in the position shown in Fig. 3. The galvanometer should deflect and immediately come to rest; the deflection represents the difference between the boiling-point of water or sulphur, and the temperature of the cold junction. Repeat for each substance at least four times, or as many times more as is necessary to reach concordant results. Limit of error, 5 per cent.

Calibration in Melting Zinc. — Put into a small clay or Hessian crucible, about one inch in diameter, and previously heated in a Fletcher or Forquignon furnace to redness, a lump

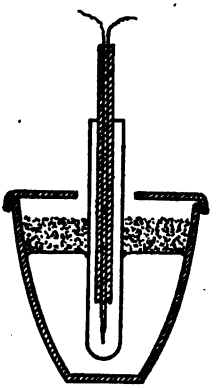


Fig. 4. — Apparatus for Determining the Melting Point of Zinc.

Scale 1 = 3

of metallic zinc about ¾ of an inch cube. Cover the zinc with powdered charcoal, cover the crucible with a perforated cover as shown in Fig. 4, apply the blast lamp, renew the charcoal as it burns away, and ascertain by poking with a glass or clay rod (not an iron rod) when the zinc is melted. Meanwhile insert the couple as shown (Fig. 4) in a small porcelain tube closed at the end, or a hard glass tube if porcelain is not to be had. Having previously carefully heated the end of the tube to incipient redness, plunge it into the bath of zinc as soon as this has melted; the galvanometer should deflect and quickly come nearly to rest, indicating the temperature of the molten zinc. To determine the freezing point of zinc, withdraw the blast lamp,

and read and record on the sheets provided the time at which the galvanometer cross hair crosses each division of the scale, and the intervals between these successive times. When the temperature has fallen to the freezing point of zinc, its further fall will be arrested, or very greatly retarded. After the first practice, the point of arrest can be readily detected. Now re-apply the blast lamp without allowing the crucible to cool completely, and without in any way disturbing it, and record time and deflections as before. When the temperature rises to the melting point of zinc, further rise will be arrested, and the cross hair will remain stationary until all the zinc has melted. Again allow the zinc to cool, and so forth. Having obtained concordant results for your first lot of zinc, repeat with a second lot.

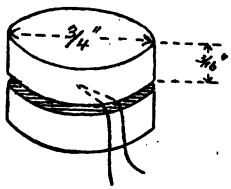


Fig. 5. — Arrangement for Determining the Critical Temperature of Steel.

Plot all the three deflections which you have now determined for the boiling points of water and sulphur, and the melting point of zinc, with temperatures as ordinates and deflections as abscissæ, and draw a smooth curve passing through them. Exhibit this to an officer of the department, who will initial it if satisfactory; if not, the experiment must be repeated.

Determination of the Critical Point of Steel of 0.80 Per Cent of Carbon. — Insert the thermo-junction between two discs of this steel, as shown in Fig. 5. Fasten the whole together with iron wire. Be very careful that the leads do not touch each other, and so short-circuit, after leaving the discs of steel. Set the two discs in a small crucible, about one inch in diameter, and heat in a Fletcher or Forquignon furnace. Regulate the flame so that the cross hair of the telescope occupies about 8 seconds in crossing each small division of the galvanometer scale. Heat to about 850° C., and record time and temperature during the heating; remove the lamp, and record time and temperature during cooling. In the column provided record the intervals between these suc-

cessive times. At the critical point $A_{1\text{-}2\text{-}3}$ there will be a considerable retardation, during both heating up and cooling down, thus lengthening one or more of these intervals. This will be made evident by comparing the successive intervals. Repeat until fully concordant results are reached. Calculate from the curve which you have plotted what the temperature of the critical point is. (It is lower and more strongly marked during cooling than during heating.)

Exhibit your results to an officer of the department, who will initial them if satisfactory. If not, the experiment must be repeated.

Additional Calibration for Students in Course 6. — Take a piece of aluminium wire one inch long, or a strip of sheet aluminium one inch long and a quarter of an inch wide, and roll that around the thermo-junction. Surround the aluminium with some thin sheet asbestos, thus completely inclosing it in a small bag or air chamber. Make the asbestos fast with thin copper wire. Imbed the whole in fine sand in a clay crucible about one inch in diameter. Heat the crucible, recording time and temperature. On approaching a low red heat the rise of temperature will be slightly retarded, and this retardation will be followed by a rapid acceleration. The point at which the acceleration begins is the melting point of aluminium.

When the retardation begins, prepare to pull the lamp away from the furnace, and as soon as the acceleration begins pull it away and record time and temperature. As the furnace cools off a retardation will be noticed which is at the melting point of aluminium. It should correspond closely to the retardation in heating.

With skill this may be repeated without disturbing the crucible.

After repeating it several times, run the temperature 100 degrees above the melting point of aluminium to watch for other retardations.

Arrangement of Squad. — Captain conducts the heating and oversees the whole.

No. 1 reads the galvanometer deflections.

No. 2 reads time, and records time and deflections.

REFERENCES: "Mesure des Températures élevées," Le Chatelier et Boudouard. P. 103, 1st paragraph; p. 104, 2d and 3d paragraphs; p. 110

(omit first 2 paragraphs); p. 111, first 3 paragraphs; p. 113, last 2 paragraphs; p. 115, 5th paragraph; p. 116, first 3 paragraphs; p. 117, first 2 paragraphs; p. 119, last 2 paragraphs; p. 120; p. 121, 1st paragraph; p. 122, 3d pargraph; p. 124, last 2 paragraphs; p. 125; p. 126, last 2 paragraphs; p. 127; p. 128; p. 129; p. 130; p. 131, first 2 paragraphs; p. 133, beginning 4 lines from the bottom; p. 134; p. 135; p. 137, 2d paragraph.

S. W. Holman, "Pyrometry: Calibration of the Le Chatelier Thermo-Electric Pyrometer," *Proc. American Academy Arts and Sciences,* XXXI (N. S. XXIII), p. 234; "Calorimetry: Methods of Cooling Correction," idem, p. 245.

Roberts-Austen, "First Report to the Alloys Research Committee of the Institution of Mechanical Engineers," pp. 4-13 of reprint. The correction for temperature of the coil and torsional resistance of the wire may be neglected.

The Author, *Trans. Am. Inst. Mining Engineers,* XXIV, pp. 798-800.

Experiment 2

To Show the Temperature at Which Steel Possesses the Hardening Power, and the Relation of this to the Magnetic Properties. — Each student does this experiment independently. Cut off ten pieces of high carbon steel wire of

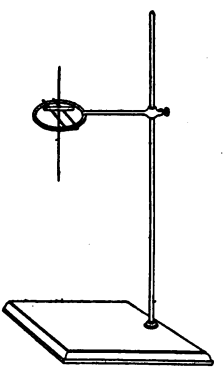


Fig. 6.

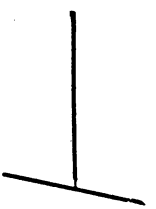


Fig. 6-a.

Fig. 6-b.

Figs. 6 to 6-b. — Arrangement of Wire, etc., for Experiment 2.

about 1 per cent of carbon, about 1-16 of an inch in diameter, and 3 inches long. Attach to the middle of each a short piece of fine platinum or manganese-steel wire as a support, as shown in Fig. 6-a. Provide yourself with a magnetic dipping needle like that shown in Fig. 6, to detect the moment when each of the

several wires under treatment becomes magnetic. This needle should be wholly uncovered, so that the wire under observation can be brought to within 1 mm. of the needle itself.

This experiment is best performed in a room as dark as is consistent with convenience of manipulation.

Heat one wire to a yellow heat in a Bunsen burner, and hold it near the needle. The wire will not be magnetic. As the wire gradually cools it will recover its magnetism and attract the needle. Note carefully the temperature, as indicated by the color, at which the magnetism is recovered, and to that end repeat the experiment. Now reheat this wire to a yellow heat and quench it in water. Try to file it: you will find it glass-hard. On trying to bend it you will find that it is extremely brittle.

Thus the sudden cooling has caused the two characteristic results of what is technically called the "hardening" of steel, for it has made the steel both extremely hard and extremely brittle. In this state it is said to be "glass hard"; and, if the percentage of carbon is high and the cooling extremely rapid, the hardened steel will scratch glass.

To determine roughly the degree of brittleness caused in this and the subsequent wires of this experiment by the sudden cooling, try to bend each wire into the shape of a hairpin, and then into the shape shown in Fig. 6-b. The wire you have already treated will break without taking any permanent bend. Fit its fragments together and attach them with gummed labels to a sheet of cardboard 6 inches square in such a way as to show that the wire has broken without permanent bend. Later do the same with all the other wires of this experiment; the angle through which each has bent before rupture is a rough measure of the ductility of its outer fibre, and thus shows to what extent the steel has been hardened by the sudden cooling.

Take a second like wire, heat it to the same high temperature, and hold it near the needle until it begins to attract the needle slightly. (In this and all subsequent operations in this series of experiments, note very carefully the temperature of the recovery of magnetism, and of the metal at the moment of quenching.) Then quench it in cold water. You will find this one also brittle and hard — i. e., hardened. Attach the fragments as before.

Heat a third wire to the same high temperature, and hold it near the needle for five seconds after it has begun to attract the

needle; then quench it in water. You will find on bending it that it is not so brittle as the preceding wires, but still somewhat brittle, and you can file it, but with difficulty — i. e., it is slightly hardened. Attach the fragments as before.

Heat another wire to the same high temperature, hold it near the needle for ten seconds after it has begun to attract the needle, and then quench it in water. You will find that this it not brittle but ductile, and that it files easily — i. e., it has not been hardened. Attach the fragments as before.

Heat still another to the same high temperature, and allow it to cool slowly in the air until it is cold enough to pick up with the fingers, but not necessarily until it is quite cold. Cool this off in water. Compare the ductility of this with that of the preceding. You will find that this, which is well annealed by the slow cooling, is somewhat, but not greatly, more ductile than the last preceding wire.

Exhibit to an officer of the department your card with the fragments of the five wires, clearly labelled, to show the degree bent by each. He will initial it and assign a mark.

This series of experiments will show that steel at a red heat is non-magnetic but has the hardening power; that it loses this hardening power and at the same time recovers the magnetic properties as it cools from a dark red heat down; and that this transformation, while rapid, is not instantaneous.

If you watch carefully in a dark room, you will see that, as the magnetic properties are returning and the hardening power is departing, the wire brightens perceptibly, owing to the heat evolved by the molecular transformation which is occurring. This brightening is known as the recalescence.

Note that in order to harden the wire at all it is necessary to heat it in the first place to a temperature considerably above that at which it loses the hardening power in slow cooling. In other words, the hardening power is acquired at a temperature higher than that at which it is lost in slow cooling.

Experiment 3

"METCALF'S EXPERIMENT." — *General Object.* — To reveal the appearance of the fracture corresponding to different temperatures to which steel has been heated.

Procedure. — A ⅜ x 1-inch bar of steel of about 0.70 per cent of carbon, about 10 inches long, is nicked on opposite faces at points ¾ of an inch apart, thus marking off on the bar a series of eight sections, each ¾ of an inch long, as shown in Fig. 7. The nicking should be done while the bar is red-hot. Number each section by stamping it with a steel stamp.

Heating. — Insert one end of this bar in the gas forge, letting the other end protrude into the air so as to keep it cool, and manage the heating in such a way as to bring the hottest section to scintillating whiteness, and so that the temperature diminishes very gradually along the bar, only the six sections nearest the hot end being visibly red-hot. In order to do this the bar will have to be moved back and forth in the forge. As soon as the bar has been so heated, withdraw it from the fire and quench it in water.

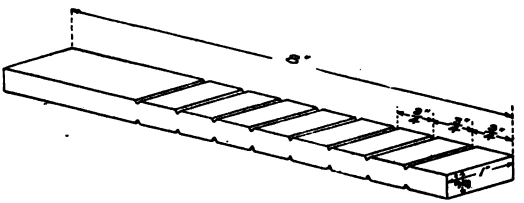


Fig 7. — Bar Nicked for Metcalf's Experiment.

Examination of Fractures. — Dry the bar extremely carefully. Wipe off all moisture very thoroughly, and then hold it above the fire till it is perfectly dry, but do not warm it above 80° C.

Set a wooden box on the floor beside the anvil, and break off each successive section by a sharp blow from a hand-hammer. Now set up the sections in order, with the fractures side by side. The fracture of the hottest section will be coarse brilliant yellowish white, while that of each successive piece will be finer and finer. That one which has been heated to the critical temperature or very little above it will be very fine or porcelanic, and those not heated to the critical temperature will be somewhat coarser and bluish.

Compare these fractures with those in Brinell's series, framed on the east wall of the furnace room.

Exhibit the assembled fractures to an officer of the department.

Arrangement of Squad for Experiment 3. — Captain — General Supervision.

Student No. 1. Break off the sections after bar has been quenched.

Student No. 2. Collect them so as to exhibit the fractures.

Students No. 3 and 4. Conduct the heating.

REFERENCES: Brinell, *Stahl und Eisen,* V, p. 620, 1885. Metcalf, *Metallurgical Review,* I, p. 245. *Trans. Am. Soc. Civ. Eng.,* XV, pp. 284, 386. Tschernoff, *Rev. Univ.,* 2d Ser., I, p. 402. Howe, "Metallurgy of Steel," pp. 172-182.

Experiment 4

DROP TESTS TO SHOW THAT OVERHEATING EMBRITTLES STEEL, AND THAT "REFINING" RESTORES ITS DUCTILITY.

Summary:

Test Bars 1 and 2 without preparation.
" " 3 after overheating to whiteness.
" " 4 after overheating to whiteness, and refining and cooling in lime.
" " 5 after overheating to whiteness and refining.
" " 6 after overheating to a yellow heat.
" " 7 after overheating to a yellow heat and refining.

The purpose of this experiment is to show that heating steel far above its critical range, even if followed by slow cooling, embrittles it, and the more so the higher it is thus heated; and also to show that the brittleness thus caused can be removed by simply reheating the steel to the critical temperature, and cooling slowly thence.

Take seven bars, ⅜ inch square and 9 inches long, of high carbon steel, preferably containing at least 1 per cent of carbon, and number them consecutively from 1 to 7, stamping the number on each end of each bar.

Bars 1 and 2. — Test these without any preparation by setting them in the notch in the top of the swage of the drop-testing apparatus, Fig. 8, and dropping the ram on each of them from the full height of the apparatus. Record the angular deflection after each blow as determined by placing the bar on a protractor; note also the total number of blows needed to break the bar and the total deflection. The blow of the ram should not be delivered directly on the bar itself, but on the fuller which is provided for the purpose. The fuller should be placed on the middle of the

length of the bar to receive the blow of the ram. Record your results in the tabular form shown on page 17.

Bars 3, 4 *and* 5.—Heat these to a white heat (about 1200° C.) in the gas forge, but do not heat them so highly that they scintillate, or that the scale or coating of iron oxide on them melts. Then set them aside to cool in the air, supported at both ends, and separated from each other so that they cool at substantially like rates.

As soon as they are so cool that you can bear your hand on them for a quarter of a second, quench them in water to gain time. This quenching, as shown in Experiment 2, does not in itself greatly affect them.

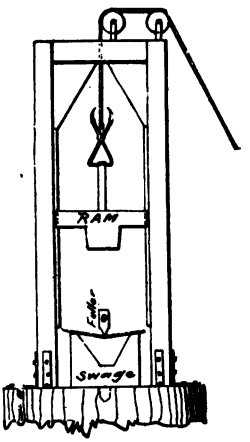


Fig. 8. — Drop-Testing Machine.

Scale 1 = 14.

Now test Bar 3 in the way in which Bars 1 and 2 have just been tested. It will bend much less, thus showing the embrittling effect of the heating to a white heat.

Before proceeding with Bars 4 and 5, repeat Experiment 2, to refresh your idea of the temperature at which the hardening power is acquired during heating. This is the temperature at which "refining" occurs.

"Refine" Bars 4 and 5 by reheating them in the forge to this critical temperature, — i. e., about 750° C. In order to cool Bar 4 very slowly, put it in powdered lime; its further treatment will be described below. In the meantime, allow Bar 5 to cool in the

air and then in water, in the same way as after the first heating, and then test it. It will bend much further before breaking than Bar 3, thus showing that refining restores the ductility removed by overheating.

Compare the fracture of 5 and 3. That of 3 is coarse, brilliant, and yellowish, while that of 5 is fine, corresponding respectively to A and C of Brinell's series, framed on the east side of the furnace room. Examine his series.

Bars 6 and 7. — Heat these like 3, 4 and 5, but not so hot, to only a moderate yellow heat, 950° C., yet well above the critical temperature, cool them in the air like 3, 4 and 5.

Test 6 as before, without any further treatment. It will bend farther without breaking than Bar 3, which had been heated to whiteness, but not so far as Bar 5, which had been refined. Compare its fracture with those of 3 and 5; it will be intermediate, Brinell's B.

Refine 7 as you refined 5, and test it. Its ductility, like that of 4, will have been restored by the refining. Examine its fracture; it will be fine, Brinell's C.

Bar 4. — Now return to Bar 4, which meanwhile has been cooling slowly in lime. It can safely be drawn after staying in lime for an hour, even if it then be too hot to touch with the hands. Cool it in the air till so cool that you can bear your hand on it for a quarter of a second, and then cool it in water to save time.

Test it like the others. It will bend even farther than bars 5 and 7, showing that the very slow cooling gives even greater ductility than the moderate slow cooling in the air. Its fracture, however, will be nearly the same as that of 5 and 7.

Moral. — Whenever you find a piece of steel which is unduly brittle and breaks with a coarse brilliant fracture, the probability is that, in heating for annealing or some other purpose, it has reached an injuriously high temperature, and has not subsequently been refined.

Arrangement of Squad for Experiment 4. — Captain — General Supervision: students Nos. 1 and 2, conduct the heating of the several bars; student No. 3, attend to the cooling, whether in water, air or lime; student No. 4, conduct the drop-tests; student No. 5, measure the deflections and make all records.

TABLE 1. — *Experiment* 4

Bar Number	Angular deflection after each blow	Total number of blows	Total deflection

Present this table to the officer of the department in charge, who will initial it if satisfactory. If not, the experiment must be repeated.

Experiment 5

MICROSCOPIC METALLOGRAPHY. FIRST AFTERNOON. EXAMINATION OF SILVER-COPPER ALLOYS. — (1) *Rough Polishing.* — The officer in charge will make a demonstration of the method of rough polishing, using the polishing machine; and each student in turn will also rough-polish a piece of the steel to be used on the second afternoon, using first the coarse emery wheel, and then a leather-covered wheel coated with No. 100 emery. While awaiting his turn at the machine, and after completing his work there, he will proceed with the fine polishing next described.

(2) *Fine Polishing.* Each student will polish finely two sections of copper-silver alloys, until no scratches are visible to the naked eye. This is effected by first rubbing the section upon emery papers of different degrees of fineness, known as 0, 00, 000 and 0000, respectively, stretched over a firm smooth backing. These should be used in the order named. The rubbing will detach some of the powdered emery from the emery paper; avoid getting this detached emery upon the board and the adjoining papers by careless rubbing. The final polishing to remove all scratches is then given by holding the section against a revolving felt buff, covered with rouge.

(3) *Heating.* — Heat the polished sections very gently in a Bunsen lamp flame, cooling and examining them at short intervals under the microscope, and carry the heating to such a point that the copper in the alloy is distinctly colored by acquiring a coating of oxide.

The copper-silver alloys consist either of a solid solution of

a small quantity of one metal in an excess of the other, or else of the eutectic, Levol's alloy (copper 28 per cent, silver 72 per cent), alone or with an excess of either argentiferous copper or cupriferous silver. Of the alloys given to the several students for polishing and heating, some will be Levol's alloy, some Levol's alloy with an excess of copper, some Levol's alloy with an excess of silver. After successfully preparing his own specimens, each student will make sketches showing their appearance under the microscope, and exhibit these to the officer in charge, who will initial them if satisfactory. He will also compare his sections under the microscope with those prepared by the other students of his squad, and with the framed series of photomicrographs of these alloys.

SECOND AFTERNOON — EXAMINATION OF HYPO-EUTECTIC STEEL. (4) *Etching.* — Each student will polish finely two pieces of steel of about 0.14 per cent of carbon, and etch them with nitric acid to develop and reveal the outlines of the grains of ferrite, of which it is chiefly composed. Immerse the finely polished section for from 6 to 15 seconds in nitric acid of 1 : 5; wash off with water, then with alcohol, and dry. The acid etches the points between the grains of ferrite more deeply than it etches the polygons of ferrite themselves. It now remains to make this effect of the etching evident, by rubbing gently just so much that the blackness caused by the etching may be removed from the polygons themselves, making them white, but not from the deepened joints between them, thus leaving these joints dark. To this end, examine under the microscope, polish very gently on chamois skin, and reëxamine; continue polishing and examining until the polygonal grains of ferrite look white, and the joints of pearlite which surround them look dark. If the polygons are still dark, you have not polished enough; if the whole surface is white and there are no dark joints dividing the mass up into polygons, you have rubbed too much, i. e., you have rubbed the joints bright. In this case you must again darken the joints by reëtching for a few seconds, and again begin rubbing gently on chamois as before. This etching and rubbing must be kept up until you have succeeded in rubbing just enough to make the polygons white, but not enough to make the joints between them white.

Compare the appearance of your section under the microscope with the series of photomicrographs of the same steel, simi-

larly prepared for your guidance. Prepare a sketch of your section, and exhibit it to the officer in charge, who will initial it.

REFERENCES: A. Sauveur, "Microscopical Examination of Iron and Steel," *Engineering and Mining Journal,* LXIV, p. 215.

Stead, "A Practical Lesson in the Preparation of Metal Sections for Microscopic Examination," *Proceedings South Staffordshire Institute,* Session 1896-97, XII, p. 2.

A. Sauveur, "Mechanical Uses of the Science of Metallography," *Engineering Magazine,* XVII, p. 980.

F. Osmond, "Méthode Générale pour l'Analyse Micrographique des Aciers au Carbone," *Bulletin Société d'Encouragement pour l'Industrie Nationale,* May, 1895.

Sir Wm. Roberts-Austen, "Microphotography of Steel Rails," *Institution of Civil Engineers,* January 17, 1899.

Osmond, "La Métallographie Considérée comme Méthode d'Essai," *Baumaterialienkunde,* II, No. 4.

Stead, "Practical Metallography," *The Metallographist,* III, p. 220.

Experiment 6

OXIDIZING ROASTING OF CUPRIFEROUS IRON PYRITES. — *General Plan.* — A sulphide copper ore is to be roasted, and the progress of the roast ascertained by taking frequent samples, and determining in each (1) how much of the copper has been converted into sulphate, (2) how much into oxide, and (3) how much undecomposed copper sulphide remains. As the roast progresses, and the ore is lightened by the conversion of the heavy iron pyrites of which it chiefly consists into the lighter form of iron oxide, the percentage of copper in the ore increases.

The copper present as sulphate is to be separated by washing with hot water; that present as oxide by attacking the residue with hydrochloric acid; and that present as sulphide by attacking this second residue with nitric acid.

The importance of ascertaining how much copper sulphate is present at each stage arises from the fact that, if the ore were to be smelted in a cupola furnace, part of the copper sulphate would be reduced to copper sulphide. Further, in certain wet processes copper sulphate behaves differently from copper oxide.

Keep the temperature low at first, to avoid fritting. Toward the end it may be raised to decompose the copper and iron sulphates.

Ferrous sulphate decomposes spontaneously when heated to

about 590° C., and cupric sulphate when heated to about 655° C. Silver sulphate decomposes thus at about 870° C. The decomposition leaves iron and copper in the condition of oxide, but silver in large part in the metallic state.

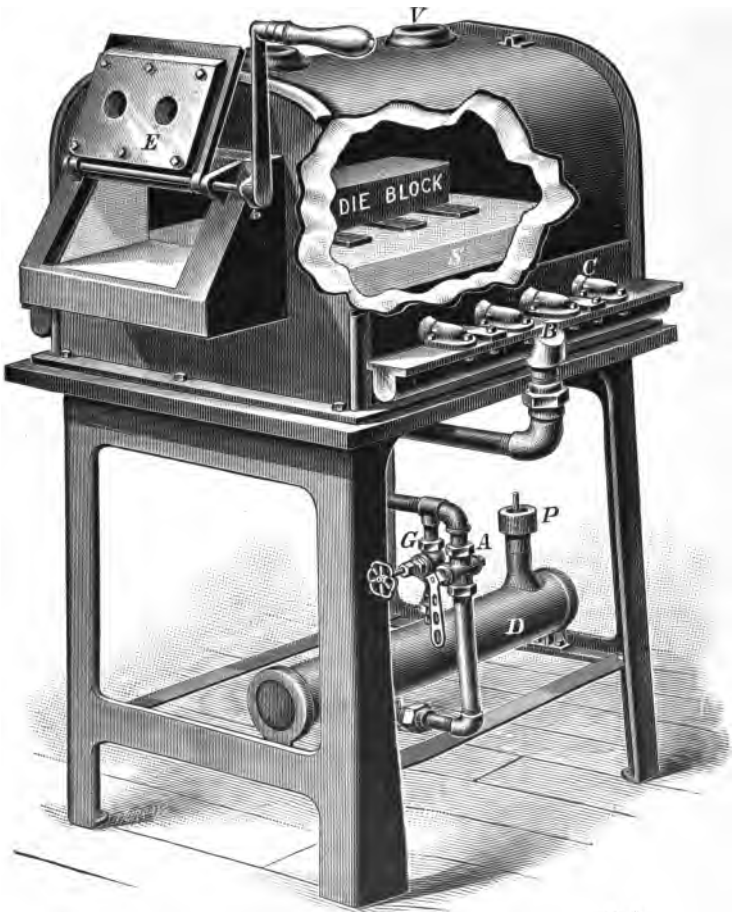


Fig. 9.— Oven Furnace (American Gas Furnace Co.)

Legend: *A*, air valve.
B, distributing channel.
C, burners.
D, equalizing receiver.
E, door.
G, gas valve.
P, weighted valve.
S, fire brick hearth, dividing oven into combustion chamber below and heating chamber above.
V, vent.

A definite task is assigned to each student, as explained in the detailed instructions.

Roast in the "oven furnace" (Fig. 9), in an iron pan, two pounds of cupreous pyrites, ground extremely fine (to pass a screen of 16 meshes to the linear inch) to hasten the roasting. Roast the same lot of ore on two successive afternoons so as to get a longer effective roast. On the first day heat moderately until the sulphur begins to burn; then check the heat until the blue flames disappear. As soon as this has occurred, raise the temperature slowly so as to reach 550° C. at 5 P.M. On the second day begin to roast at 450° C., and raise the temperature slowly so as to reach 850° C. at 5 P.M.

Place the thermo-junction of a Le Chatelier pyrometer in a narrow porcelain tube closed at its lower end, and imbed this end in the roasting ore.

Record. — The results thus obtained must be recorded (1) in the printed table provided for the purpose, (2) graphically, and (3) on the blackboard. Submit all records to an officer of the department, who will initial them if satisfactory.

Arrangement of Squad. — 1. Rabble the roasting ore.

2. Read the pyrometer and record the readings on the blackboard.

3. Take the samples, leach them with water, assay, and record the results on the blackboard.

4. Leach the samples with hydrochloric acid, assay, and record on the blackboard.

5. Leach with nitric acid, assay, and record on the blackboard.

6. Captain — General Supervision.

Detailed Instructions. — Captain.

See that the furnace is started at 2 P.M.

See that the roasting ore is properly stirred.

See that the assaying of roasting ore is carried out promptly, and recorded properly.

(Student 1.) *Rabbling.* — While the sulphur is burning off during the early part of roasting, stir thoroughly every 10 minutes and just before each sample is taken. After the blue flames have disappeared stir every 15 minutes until the middle of the second afternoon; thereafter stir every 10 minutes until the roasting is completed. When stirring, be sure to break up all crusts.

Keep the peep-hole closed as much as possible, to avoid lowering the temperature and to prevent the escape of sulphur dioxide (SO_2). Keep the pyrometer tube well covered with ore, so that the galvanometer readings may correctly represent the temperature of the ore.

(Student 2.) *Controlling the Temperature.* — From the curve of the thermo-electric couple used, determine approximately the galvanometer readings equivalent to the desired temperatures, and make frequent galvanometer readings. Regulate the gas supply so that the temperature shall increase uniformly, without exceeding the limits given in the general directions. The temperature should be observed before each sampling, and recorded in degrees Centigrade, on the blackboard and graphically on co-ordinate paper.

(Student 3.) *Sampling and Determining Sulphate.* — Take a sample, after thorough rabbling, every 30 minutes. Of this, put 3 grammes into a 4¼-inch casserole containing 100 c.c. of boiling water. Heat to boiling again, and decant the solution carefully into a 500 c.c. round flask. Add another 100 c.c. of boiling water to the residue in the casserole and decant this wash water into the same flask. Mark the casserole containing the residue with the running number of the sample, and turn it over to student No. 4. Add 5 c.c. of concentrated nitric acid to the contents of the flask, and heat them to boiling for 5 minutes; then add ammonia in slight excess, — enough to redissolve any copper hydrate formed — cool quickly and titrate in the flask with standardized potassium cyanide solution, without removing any precipitate which may form. Record the result opposite the proper number in the column headed "Sulphate," and calculate "Formation of Sulphate per hour." (*Note.* — Do not allow the samples to accumulate. Run each assay as promptly as possible, with several under way at once if necessary.)

(Student 4.) *Determining Copper Oxide.* — Dissolve the oxides in the casserole as received from student No. 3, by adding 150 c.c. of dilute hydrochloric acid (1 part concentrated acid to 9 parts water) and boiling 1 minute. Allow the residue to settle, and decant the solution into a 500 c.c. round flask. Wash the residue by adding 50 c.c. of boiling water and decanting it into the same flask. Give the residue in the casserole to student No. 5. Add to the solution in the flask 5 c.c. of concentrated nitric acid

and boil for 5 minutes; then add to the boiling solution ammonia in slight excess — enough to redissolve any copper hydrate thrown down, — cool, and titrate in the flask with standardized potassium cyanide solution, without removing other precipitates which may be present. Record the result opposite the proper number in the column headed "Oxide." (*Note.* — Do not allow the samples to accumulate. Run each assay as promptly as possible, with several under way at once if necessary.)

(Student 5.) *Determining Copper Sulphide.* — To the residue in the casserole as received from student No. 4, add 25 c.c. of concentrated nitric acid, and boil for 10 minutes. Wash both solution and residue into a 500 c.c. round flask with 100 c.c. of hot water; heat to boiling, add ammonia in slight excess, — to dissolve any copper hydrate formed, — cool, and titrate with standardized potassium cyanide solution in the flask, without removing the residue or any precipitates. Enter the results opposite the proper number in the column headed "Sulphide," and calculate "Decrease of Copper Sulphide per hour." (*Note.* — Do not allow the samples to accumulate. Run each assay as promptly as possible, with several under way at once if necessary.)

Supplies Needed for Copper Roasting. — (1) Two pounds cupreous pyrites, preferably not under 5 per cent copper, crushed to pass a screen of 16 meshes to the linear inch.

(2) A scoop for sampling, 10 c.c. capacity.

(3) An iron pan to hold the ore during roasting (20 in. x 4 in. x ¾ in.).

(4) A rabble for stirring.

(5) A connection with a portable Le Chatelier pyrometer. Place the couple in a porcelain tube and imbed it in the roasting ore.

(6) A blackboard upon which to put down the results of analysis as fast as reached. Record on it Time, Temperature, Copper as Sulphate, as Oxide and as Sulphide. (See Table 2.)

(7) Reagents and apparatus:

Hydrochloric acid, nitric acid, and ammonia (all concentrated);
standardized potassium cyanide solution;
6 casseroles (4¼ inch);
12 flasks (500 c.c. round);
3 burettes with stands;

3 wash bottles;
6 stirring rods;
a heating plate;
small gummed labels.

TABLE 2. — *Experiment* 6

*Record of Results by*_____ *Captain of Squad No.*_____

Serial Number	Time				Temperature		Composition of Ore				Rate of Oxidation	
	Local		From beginning of roast		Galvanometer deflections	Corrected temperatures °C	Copper as					
	Hours	Minutes	Hours	Minutes			Sulphide	Oxide	Sulphate	Total	Decrease of Sulphide per hour	Formation of Sulphate per hour

Tabular form of record for Experiment 6.

Experiment 7

DESILVERIZATION OF BASE BULLION, PARKE'S PROCESS.* — *Principles Involved.* — The process depends upon the fact that when metallic zinc is mixed with molten base bullion (auriferous or argentiferous lead), the zinc alloys with the gold, copper, and silver, in this same order. The resulting alloy has a higher melting point than the base bullion. At a temperature below its own melting point it is only very slightly soluble in molten lead, especially in lead already saturated with zinc. It is lighter than the base bullion or the desilverized lead. If, therefore, the mixture be cooled below the melting point of the zinc alloy, this will separate out and float on the surface of the lead as a mushy crust, which can be removed by skimming; and the lower the temperature (provided it be above the melting point of the

* The details of this experiment were worked out by Mr. W. A. Bentley.

lead) the more complete is the separation. By three successive treatments, involving the addition of 2.5 per cent of zinc in all, the silver contents of base bullion containing 200 oz. per ton should be reduced to less than 0.5 oz. per ton, and the silver and gold should be concentrated in zinc crusts the total weight of which should not exceed 15 per cent of that of the original bullion.

REFERENCE: Hofman, "Metallurgy of Lead," 5th Ed., p. 427 et seq.

First Afternoon. — Fuse and skim the base bullion and add the first zinc. Remove the first crust.

Second Afternoon. — Add the second and third zincs. Remove the corresponding crusts.

Subdivision of Squad. — The squad will divide into two sections. Section 1 will be composed of the first Captain and Nos. 2 and 3; section 2 will be composed of the second Captain and Nos. 4 and 5. Each section will work independently of the other.

Arrangement of Squad. — Captain — General oversight; weighing base bullion, dross, zinc, and crusts, calculating silver contents, and recording results on blackboard.

Student No. 2 (or No. 4). Managing furnace and kettle, adding zinc, removing crusts, and taking samples.

Student No. 3 (or No. 5). Assaying.

Supplies Needed. — 20 lbs. base bullion, 1 lb. zinc, 1 iron melting pot (7 in. in diameter and 4½ in. deep), 1 iron skimmer (perforated), 1 iron ladle, 1 iron mould for samples, 3 iron pans for crusts, 10 cupels, 5 scorifying cups, test lead, ore and button balances, scales for weighing base bullion, Forquignon furnace, high temperature thermometer.

Detailed Instructions. — Captain — See that the furnace and materials are in readiness. Weigh out 20 lbs. of base bullion containing about 200 oz. silver to the ton, or corresponding quantities of soft lead and silver. Weigh out

"First zinc" ..... 8 oz.
"Second zinc" ..... 4 oz.
"Third zinc" ..... 4 oz.

and deliver to student No. 2 as required. Receive from student No. 2, weigh, and record weights of the following: Dross, First Crust, Second Crust, Third Crust, and Desilverized Lead. Obtain from student No. 3 results of assays, and record them. Make all necessary calculations for the record, which should be kept in the form given, Table 3.

(Student 2.) *Managing Furnace and Kettle. First Afternoon.* — Melt the base bullion in an iron melting kettle over the Forquignon furnace, keeping the kettle covered with a sheet of asbestos. When melted, stir thoroughly, and with the skimmer remove any dross, which should then be delivered to the Captain of the squad for weighing. Take two assay samples of the base bullion of approximately ½ assay ton each, and deliver to student No. 3. Then add the "first zinc," — to be provided by the Captain, — heat, and stir until it is wholly melted and thoroughly mixed with the lead. Allow the lead to cool rather slowly, working the crusts that form on the sides of the kettle toward the centre, until the temperature of the base bullion, as measured by the thermometer, has fallen to 350° C. Then with the previously heated skimmer remove the mushy alloy or "crust" which rises to the surface, with as little adhering lead as possible. Collect the crust in an iron pan; mark it "first crust," and deliver it to the Captain. Continue until the crust ceases to separate, or the base bullion is near its freezing point; then reheat the remaining base bullion, mix it thoroughly, and take assay samples as before; turn off the gas, and allow the base bullion to cool in the kettle.

Second Afternoon. — Remelt the contents of the kettle, add the "second zinc," and proceed as after the addition of the first zinc. Mark the skimmings "second crust," then reheat the kettle, stir its contents, and take assay samples as before. In like manner add the "third zinc," and take the "third crust" and samples for assay; then ladle the now desilverized lead into a mould, allow it to cool, and deliver it to the Captain.

(Student 3.) *Assaying.* — Assay by fire the samples taken by student No. 2, which should be as follows:

Base bullion before the addition of the first zinc.

Base bullion after the removal of the first crust.

Base bullion after the removal of the second crust.

Desilverized lead.

Report the results to the Captain, who will record them on the blackboard. (*Note.* — The assay samples taken after adding zinc to the base bullion should be scorified before they are cupelled.)

Each student will hand to the officer in charge, in a sealed envelope, his own independent explanation of the principle of the Parke's process, and a statement of the thoroughness of the desilverization brought about by each of the additions of zinc.

TABLE 3

Record of Results by ..... *Captain of Squad*

	Time	Weight	Assay	Silver Contents
1. Original base bullion	....	....	omit	omit
2. Dross	....	....	"	"
3. Base bullion after drossing	....	*	....	.....
4. Assay sample	....	....	....	
5. First zinc	....	....		
6. First crust	....	....	*	*
7. Base bullion after removal of 1st crust	....	*	....	.....
8. Assay sample	....	....	....	
9. Second zinc	....	....		
10. Second crust	....	....	*	*
11. Base bullion after removal of 2d crust	....	*	....	.....
12. Assay sample	....	....	....	
13. Third zinc	....	....		
14. Third crust	....	....	*	*
15. Desilverized lead	....	....	....	.....
16. Assay sample	....	....	....	

* Calculate by difference.

Tabular form of record for Experiment 7.

Experiment 8

TEMPERING. — Harden ten pieces of steel wire of about 1 per cent carbon and 1-16 of an inch in diameter, each about 4 inches long. Brighten them with sandpaper and temper them by heating them carefully, for instance, by holding near a piece of red-hot iron. Watch the progress of the tempering as indicated by the successive oxide tints that form, first straw and last blue.

Prepare a succession of wires showing this succession of oxide tints. Also heat two wires hotter, one to barely visible redness and one to full redness, and allow all to cool slowly in the air.

Now ascertain how the tempering accompanies these oxide tints, by determining the transverse elastic limit of these wires. To this end suspend them between horizontal supports in the testing machine shown in Fig. 10, and load them with progressively increasing loads, recording the deflection corresponding to each load, and the ultimate load. Plot curves of the results with stress

as ordinate and strain as abscissa. This will indicate the progress of the removal of the hardening (1) by the slight reheating or tempering, and also (2) by the annealing caused by heating two of the wires to redness.

Compare the properties of the several wires, noting in par-

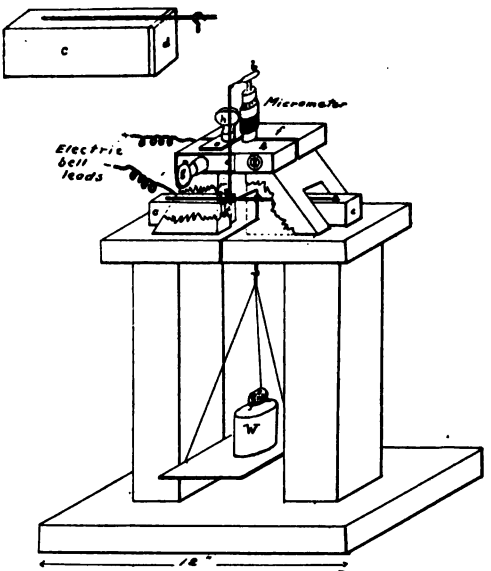


Fig. 10. — Testing Machine for Experiment 8.

Legend:

a, the wire under test.

b, continuation of the rod by which the load is applied. The deflection is measured by bringing the micrometer into contact with *b*, the moment of contact being determined by electric bell contact.

c, movable blocks which support the wire under test.

d, metal face to block *c*.

e, *f*, jaws for holding micrometer.

g, screw to draw jaws together.

h, screw for centering micrometer when grasped in jaws.

ticular (1) the modulus of elasticity as roughly indicated by the deflection under the earlier loads; (2) the elastic limit, as indicated by the departure from Hooke's law; (3) the ultimate load, that under which the wire gives way; and (4) in case of those wires which break, the ductility, as shown by the angle which the fragments make when fitted together.

Test the several wires with a file, to learn how the removal of the hardness, which sudden cooling had caused, accompanies the other changes due to tempering.

Each student performs this experiment independently.

Experiment 9

Removal of the Effects of Overstrain by Reheating. — Take twelve 4-inch pieces of wire exactly similar to that used in the last experiment, except that it has been hard-drawn and has not been annealed. Test two of them transversely in the hard-drawn state, as described in the last experiment.

Heat four of them to successively higher temperatures, judging by the oxide tints which form on the surface. Heat them by holding them near a piece of red-hot iron. In order that the heating should be uniform the wire should be moved about and rotated.

Heat two of them in boiling water, two in boiling linseed oil, and two in melting lead. In each case cool them slowly.

Test all twelve wires transversely and plot the results with stress as ordinate and strain as abscissa.

This experiment will show how the removal of the effects of cold-working or "overstrain," or in other words the annealing of cold-worked iron, progresses as the temperature rises.

Each student performs this experiment independently.

Experiment 10

Hardening is a Function of the Carbon-Content. — Take two pieces of high carbon steel wire (0.50 to 1 per cent carbon), about 1/8 of an inch thick and three inches long, two of medium carbon steel wire (about 0.25 per cent carbon), two of low carbon steel wire (about 0.10 per cent carbon), two of wrought-iron wire, and two of copper wire. Prepare a freezing mixture with salt and snow, or salt and crushed ice.

Heat one of each of these wires in a blast lamp as hot as you can heat it, or in case of the copper wire, as hot as you can heat it without melting it. In doing this hold the wire with a pair of tongs or with a piece of fine iron wire.

Quench in cold water, or in the freezing mixture, all the wires thus heated.

Determine the ductility of each of these wires, and also of the untreated wire of each class, by bending the wire until it breaks or until it is bent double as in Fig. 6-b. In doing this, one end of the wire should be held in a vise. Fit the fragments of each wire together and fasten to a card so as to show the angle bent before rupture. Show the card to the proper officer, who will assign a mark. Also test the hardness of the steel wires by means of a file and by trying to scratch glass with them.

You will find that the high carbon steel wire when hardened will scratch glass, is not cut by a file, and is extremely brittle; that the medium carbon wire, while very brittle, is less so than the preceding, and while greatly hardened can yet be filed, and will not scratch glass; that the soft steel, wrought iron and copper wires have hardly been affected at all. From your results enunciate your inferences as to the relation between the hardening power and the carbon-content.

GROUP 2. — PYROMETRY AND CALORIMETRY

Experiment 11

SIEMENS' WATER PYROMETER. — *The Method of Mixtures.* — Read the description and manner of use — Journal Iron and Steel Institute, 1871, I, p. 51 and plate 4 (not numbered in original) or description of "The Method of Mixtures" as applied to determining specific heat, pages 436 to 438, Ganot's "Physics" (14th edition of Atkinson's translation, 1893).

Principle. — In this method the temperature is determined by heating a solid copper cylinder to it, and then ascertaining through how many degrees this cylinder in cooling will raise a known quantity of water in which it is next immersed. Knowing the thermal capacity (1) of the copper cylinder, and (2) of the water and the vessel containing it, the initial temperature of the copper cylinder is found by the formula:

$$x^\circ = \frac{(\theta - t)^\circ (W + 'wc')}{wc} + \theta^\circ$$

in which

W = weight of water in grammes

t° = initial temperature of water (centigrade)

θ° = corrected maximum temperature of mixture (see Experiment 12 for cooling correction)

w = weight of substance (copper cylinder)
c = specific heat of substance (copper cylinder)
w′ = weight of vessel (calorimeter)
c′ = specific heat of vessel (calorimeter)
x = initial temperature of substance (copper cylinder).

Procedure. — Fill the calorimeter with water to the mark on the inside, and adjust the zero of the brass scale to the level of the mercury in the thermometer. This weight of water corresponds to the weight of the copper cylinder, so that the actual rise of the mercury will indicate directly on the adjoining brass scale the temperature of the copper cylinder at the instant when it is plunged into the water of the calorimeter.

Now determine the temperature of a muffle furnace which, together with a 2-inch scorifier standing within it, has been brought to a dull red heat. To this end set the copper cylinder in this scorifier, and leave it there for 15 minutes, keeping the temperature of the muffle as nearly constant as you can, and leaving its door closed. During this time the cylinder will very nearly reach the temperature of the muffle itself.

Remove the cylinder from the furnace, using a pair of tongs the ends of which have been preheated so as to lessen the amount of heat which they will remove from the cylinder by conduction, and drop it into the calorimeter, making the transfer as rapid as possible so as to lessen the loss of heat in transit. As soon as the cylinder is in the calorimeter, cover the top with a small piece of cardboard and note the highest point reached by the thermometer.

Check the results with the Le Chatelier (thermo-electric) pyrometer.

Experiment 12

MAHLER'S BERTHELOT CALORIMETER.* — *Principle of the Apparatus.* — We determine the calorific power of a fuel by noting the quantity of heat, Q, evolved on burning a known weight of it with oxygen gas while enclosed in a hermetically sealed bomb, immersed in a vessel containing a known quantity of water. This heat imparts itself to the bomb and to this water jointly;

* See Berthelot, "Traité Pratique de Calorimétrie Chimique"; *Le Génie Civil,* January 23, 1892; J. Struthers, *School of Mines Quarterly,* XVI, p. 209; *Bulletin de la Société d'Encouragement pour l'Industrie Nationale,* June, 1892; *Journal of the Iron and Steel Institute,* No. 1, 1892; *Mineral Industry,* Vol. I, 1893.

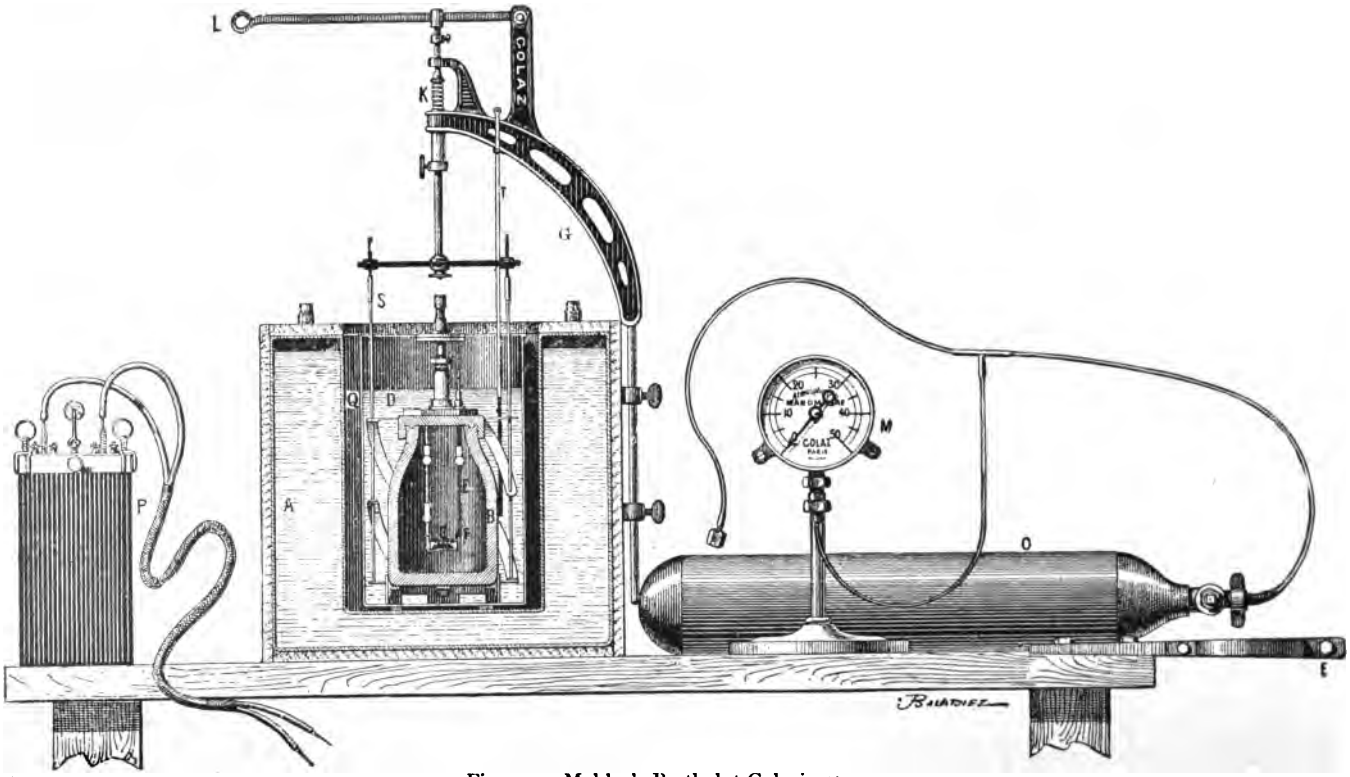


Fig. 11.— Mahler's Berthelot Calorimeter.

Legend:

A, isolating vessel; B, the calorimetric bomb of soft steel, enameled on the inside surface; C, the platinum dish to hold the combustible; D, the calorimeter vessel filled with water; E, the isolated electrode; F, the iron wire which ignites the combustible; G, the support for the agitating apparatus; K, the agitating mechanism; L, the arm for imparting power to the mechanism; S, the agitator; M, the manometer; O, the cylinder of oxygen under high pressure; P, the chromate battery for heating the iron wire in the bomb; T, the thermometer; Q, the calorimeter brass cylinder.

and the number of degrees by which their temperature is thus raised is carefully measured. Then, knowing the number of calories needed to raise the bomb, and also the water, through one degree Centigrade, we calculate the heat which the combustion has evolved; and knowing the weight of the fuel burnt we calculate the calorific power of a unit of fuel.

Corrections must be made for the escape of heat during the determination, and for the heat evolved by the combustion of nitrogen to nitric acid, etc.

Preparation of the Fuel. — If this be bituminous coal or other fuel liable to yield tar or soot on burning, it should be compressed into a firm cake so as to reduce this tendency to a minimum. Compress a little over one gramme of the fuel into such a tablet by means of the mould and screw press provided.

The Apparatus. — The calorimeter, Fig. 11, consists essentially of the tightly sealed very strong bomb, B, containing a platinum cup, C, in which the tablet of fuel is burnt, and surrounded by a known mass of water, D, in a thin brass cylinder, Q. This water is stirred by means of the stirrer, S, and its rise of temperature, due to the heat evolved by the burning fuel, is read by the delicate thermometer, T. The foregoing parts constitute the calorimeter proper.

The escape of heat from the outer surface of the calorimeter should be (1) slow, and (2) regular and thus measurable. To make it slow the outer surface of the cylinder, Q, should be (a) highly polished, and therefore nickel-plated, and (b) surrounded with an air space, and this in turn with a felted, double-walled tank containing a large mass of water, A, at constant temperature. The great mass and external felting of this outer vesselful of water, and the consequent slowness of its change of temperature, cause the escape of heat from the calorimeter itself, Q, to be regular, so that the second of the above conditions may be fulfilled; to this same end the water in both vessels should initially be above the dew-point, lest dew condense on either of them,*

* The essential point here is that the water should be above the dew-point of the room; but as the dew-point is not usually known, a convenient way of insuring this is to have the water in the tank slightly above the temperature of the room itself, and that of the calorimeter within 1° C. of the temperature of the room.

It is often directed that the temperature of the water be initially

and later reëvaporate, so imparting heat to the apparatus, or removing heat from it, irregularly and thus indeterminably.

Ignition.—The fuel is ignited by means of a fine iron wire previously imbedded in it, and heated to redness by a current of electricity generated by the plunge battery P, and carried to the wire by means of its supporting rod, E, and of the stem which supports the platinum cup, C.

Combustion is kept up by means of oxygen gas from the flask O, previously forced through a needle valve into the bomb under pressure, which is read by the gauge M.

Manipulation. Place the tablet of fuel in the platinum dish, C. Fasten a weighed piece of iron wire of 36 B. & S. gauge, six inches long, at one end to the rod E and at the other to the rod which supports the platinum cup, and bury part of this wire in the tablet of fuel. Screw the top of the bomb on firmly, fill it with pure oxygen at a pressure of 350 pounds per square inch,* close the needle valve, set the bomb in place, and fill the outer tank with water warmer, but not more than 3°C. warmer, than the room. Pour into the calorimeter cylinder 2512† c.c. of distilled water at a temperature not more than 1° C. different from that of the room. Stir the water in the calorimeter and read and record its temperature once a minute for five minutes, determined by a stop watch. Then pass the current through the iron igniting wire, and continue reading and recording time and temperature every minute until five minutes after the temperature has passed its maximum, noting also with care the actual maximum, and the moment at which it was reached.

---

slightly below the room-temperature, so that at the end of the experiment it will be only slightly above that temperature, with the idea of having the inflow of heat from the room into the calorimeter in the early part of the experiment approximately balance the outflow of heat from the calorimeter into the room in the latter part. Doubtless this tends to diminish the total transfer of heat between calorimeter and room; but, as a cooling correction based on direct observation must be introduced in any event, it is far better that this transfer of heat should be regular and thus measurable, than that it should be probably small.

* The oxygen should be pure, not commercial. Suitable oxygen at a pressure of 2,200 pounds per square inch, is supplied in 150-gallon cylinders by the S. S. White Dental Manufacturing Company and others.

† This quantity, added to the water-equivalent of the calorimeter itself, viz.: 488 grammes, gives a convenient number, 3,000 grammes, as the water-equivalent of the whole.

Acidimetry. — Allow the oxygen gas to escape from the bomb very gently. Wash the contents of the bomb into a beaker, and determine (1) the nitric acid formed by titration with alkaline solution, and (2) the sulphuric acid, by weighing as barium sulphate.

Corrections.—The observed rise of temperature must be corrected for the loss of heat which occurs during the experiment; and from the heat as calculated from this corrected rise of temperature we must deduct that generated in the experiment by the formation of nitric acid, because this would not be formed in burning the fuel under common industrial conditions.

Cooling Correction. — (See Fig. 12.) "On a sufficiently large scale, not less than 0.01 inch to 0.1 of smallest scale-division of the thermometer, plot points with times as abscissas, and observed corrected temperatures of the calorimeter as ordinates. A

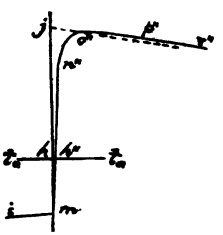


Fig. 12.

smooth line drawn through these points will have the general form of the line *imn"o"p"r"* . . . . Find carefully the maximum point *o"* of this curve. The part *p"r"* of the line will be sensibly straight. Draw through *o"* a line *o"j* parallel to *p"r"*. Then the length of the intercept *mj* on the ordinate through *m*, the time of insertion of the hot substance, will be the rise of temperature which would have occurred in the calorimeter, had no loss by cooling occurred, and gives us directly the corrected rise of temperature."*

Correction for Nitric Acid.—Using methyl orange as an indicator, titrate the nitric acid with a solution containing 3.706 grammes of anhydrous sodium carbonate per litre, so that each c.c. represents 0.0044 grammes of nitric acid, the heat of formation of which is 227 calories per gramme. Hence each c.c. repre-

* "Physical Laboratory Notes," S. W. Holman, p. 46, 1887. J. S. Cushing & Co., Boston.

sents 0.0044 x 227 = 1.0 calory, and hence from the total heat developed by the combustion we must deduct one calory for every c.c. of sodium carbonate solution.

Sulphuric Acid Correction. — The above titration, however, determines not only the nitric but also the sulphuric acid formed by the combustion of the sulphur of the fuel. As this sulphuric acid also forms in actual industrial combustion, so that the heat developed by the oxidation of the sulphur is part of the useful calorific power of the fuel, we must, in case much sulphur be present, subtract from the total number of c.c. of sodium carbonate solution neutralized, the number of c.c. neutralized by the sulphuric acid thus formed, before making the above correction. Determine the sulphuric acid, which is present as sodium sulphate, by precipitating it with barium chloride and weighing the precipitate of barium sulphate.

.0081 gm. $BaSO_4$ = .0034 gm. H_2SO_4 = 1 c.c. Na_2CO_3 solution.

$$\therefore \frac{\text{Wt. of } BaSO_4 \text{ in grammes}}{.0081} = \text{c.c. } Na_2CO_3 \text{ solution due to } H_2SO_4.$$

To correct for the iron wire deduct 1650 calories, the calorific power of iron, for each gramme of wire.

Heat-Capacity of the Calorimeter.—This may be arrived at, either by adding the products of the weight in grammes of each substance contained in it multiplied by its specific heat; or by the method of mixtures. For the Mahler calorimeter in the Metallurgical Laboratory of the Columbia School of Mines it may be assumed to be equivalent to 488 grammes of water.

Calculation of Calorific Power.—The calorific power of the fuel is calculated from the corrected rise of temperature by the following formula:

$$C = \frac{\Delta (P + P') - [1650\, p + (p' - p'')]}{P''}$$

in which

C = the calorific power of one gramme of the fuel, in calories;

Δ = the corrected rise of temperature;

P = the weight of water in the calorimeter, in grammes;

P′ = the water-equivalent of the calorimeter itself;

P″ = the weight of fuel in grammes;

p = the weight in grammes of the iron wire, and 1650 its calorific power per gramme;

p′ = the number of cubic centimeters of sodium carbonate solution neutralized by both nitric and sulphuric acids;

p″ = the number of cubic centimeters of sodium carbonate solution neutralized by sulphuric acid only.

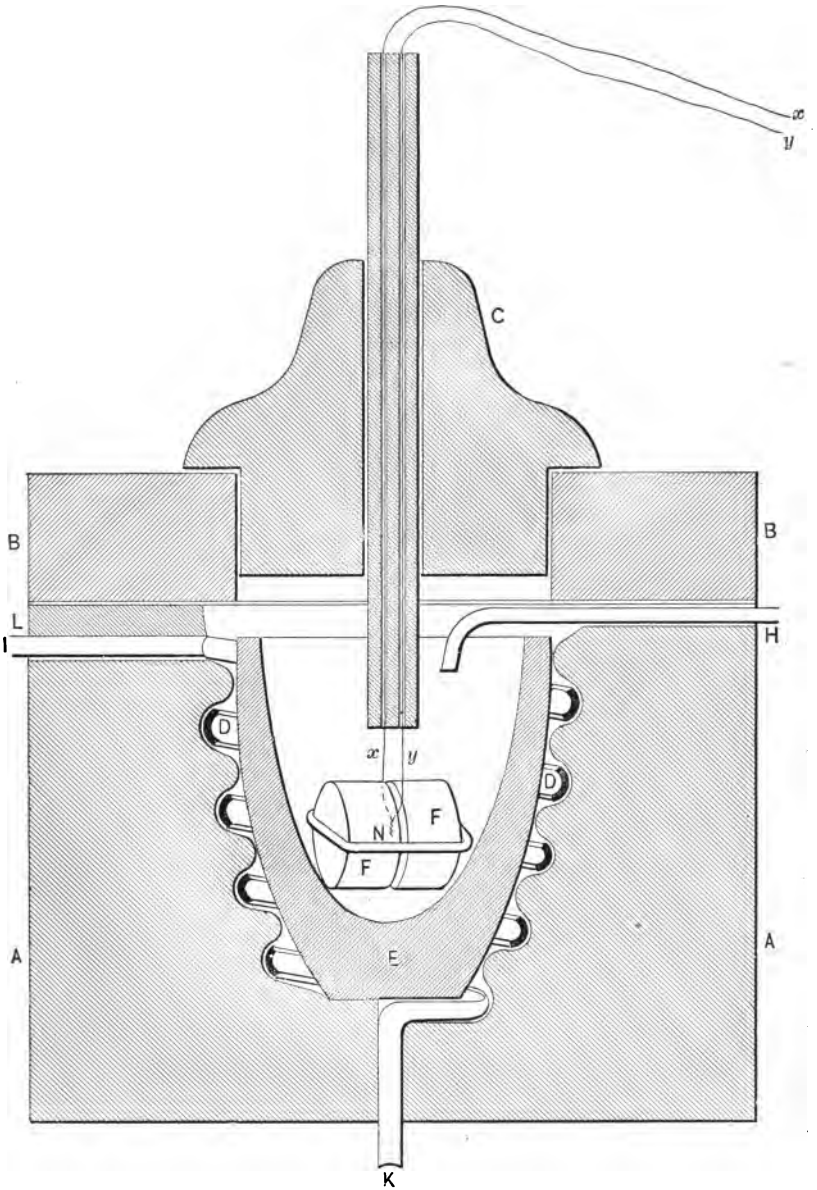


Fig. 13. — The Author's Electric-Resistance Crucible-Furnace.
(Vertical Section, Actual Size.)

Legend: AA, two semi-cylinders of magnesia; BB, magnesia cover; C, stopper (magnesia); DD_1 spiral of platinum resistance wire heated by current; E, magnesia crucible fitting loosely in spiral; FF, steel discs under test; H, pipe for introducing any desired gas; I, K, openings through which the platinum resistance wire passes; L, packing; N, thermo-junction; *x*, *y*, leads of thermo-junction.

Experiments 13 and 14

LE CHATELIER PYROMETER. — For students who have performed Experiment 1. Re-calibrate the instrument by means of the boiling points of water, naphthaline, sulphur, and zinc, and with the melting points of tin, zinc, aluminium, copper (1083° C.) and platinum. (See Le Chatelier & Boudouard, " Mesure des Températures élevées," pp. 131-139.) Besides Le Chatelier's directions, consult Holman (*Proceedings,* American Acad., Nov. 13, 1895, articles X, XI, XII and XIII).

Plot the results with temperature as ordinate and galvanometer deflection as abscissa. Plot also according to Holman's logarithmic formula.

Experiment 13

MELTING POINT OF COPPER. — A convenient way is to melt 50 grammes of pure electrolytic copper in a magnesia crucible in the electric-resistance crucible furnace, Fig. 13, keeping the copper covered with a heavy layer of powdered charcoal, and renewing this lest the metal absorb oxygen. When the temperature has risen slightly above the expected melting point of copper, lower the thermo-junction so that it dips slightly into the molten copper, and lower the temperature; when the copper begins to freeze the fall of temperature will be arrested abruptly, and the temperature will remain stationary for a long time. When it again begins to fall, and before it has fallen far, re-apply heat so as to remelt the copper, and the temperature will again remain stationary during the remelting of the copper. This manœuvre can be repeated easily, and a large number of determinations with very concordant results can be made in a very short time. But unless the copper is completely protected against oxidation concordant results cannot be had.*

Experiment 14

MELTING POINT OF PLATINUM. — In using this point for calibration, advantage is taken of the fact that the pure platinum lead of the thermo-junction itself is more fusible than the rhodio-

* The author has found it very difficult to prevent the copper from oxidizing when using Holman's arrangement.

platinum lead. The thermo-junction is accordingly heated cautiously in the oxyhydrogen blowpipe flame (see Fig. 14) until the platinum lead melts, a second observer noting the maximum deflection of the galvanometer, which should occur just as the platinum lead melts and breaks away from the junction, so that the galvanometer returns abruptly to zero.

Since the deflection of the galvanometer is that due to the temperature at the junction proper, i. e., at the point *d* where the leads separate, it is essential that this point itself should reach the melting point of platinum, 1780°. Manifestly if the platinum lead were to melt at the point *a*, the thermo-junction would itself be far below 1780°, because of the steep thermal gradient of the lead, due to the rapid conduction and radiation at so high a temper-

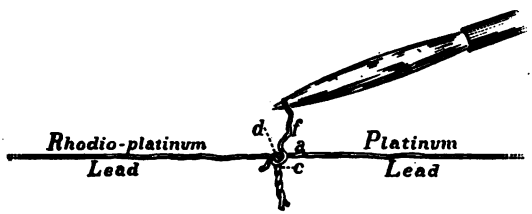


Fig. 14. — Calibration by the Melting Point of Platinum, 1780° C.

ature. If, on the other hand, the fusion were to take place at the point *c* slightly beyond the true junction, and the two leads were then to slip apart, as they readily might, the junction proper might still be much below 1780°, because of the extreme steepness of the thermal gradient. The fusion must be so managed that the junction itself reaches 1780°, for instance by winding a little spur *f* of pure platinum wire* around the junction proper, having this spur point upward, and melting it so that it runs down and covers the true junction; and then finally melting the junction itself. The little bath of platinum thus provided causes a greater length of the platinum lead to be accurately at 1780°, and thus tends to insure that the junction itself shall reach 1780°.†

* Suggested by B. Stoughton, Esq.

† Or the leads, wound into a little double corkscrew, and pointing upward, can, in like manner, be melted down so as to cover the junction with a drop.

The drop in this case is not platinum but rhodio-platinum, and consequently may be hotter than 1780°; yet the platinum lead itself will melt

Although the results are more concordant than might be expected from the apparently rather crude conditions, yet it may be well to flatten the thermal gradient of the leads by wrapping little sleeves of asbestos around them near the thermo-junction itself, so as to lessen the radiation from the leads.

Experiment 15

Optical Pyrometers of Le Chatelier and Mesuré and Nouel. — Compare Le Chatelier & Boudouard, "Mesure des Températures élevées," pp. 158-177.

The experiment consists first in calibrating these instruments by means of the thermo-electric pyrometer, and second ascertaining the accuracy of your reading of these instruments by comparison with the thermo-electric pyrometer. Place the thermo-junction of the thermo-electric pyrometer in a muffle furnace with a closed doorway, or in some other hot chamber, the temperature of which can be kept approximately uniform throughout and can be made to vary as a whole with regularity. In the door of the muffle furnace arrange an opening of any convenient shape and approximately a square inch in area. Follow the heating of the muffle furnace with the thermo-electric pyrometer and also with the two others as the temperature gradually rises to at least 1400°, getting observations at least at every 25°. Now cool the muffle furnace off, either by cutting off the supply of gas or by checking and drawing the fire, so that it will cool down slowly and regularly, and follow the cooling in the same way with all three instruments. Record observations for at least every 25°.

---

as soon as it reaches 1780°, and, if it springs away from the junction as soon as it melts, 1780° will be the maximum temperature which the galvanometer will indicate. Only, in case the platinum lead did not spring away immediately then, but were held so that it was connected with the rhodio-platinum lead through a drop of melted platinum hotter than 1780°, the temperature represented by the galvanometer deflection might be above 1780°.

Even if the junction proper be the part most highly heated, so that the galvanometer thus records the highest temperature reached, that temperature may not be strictly the melting-point of platinum, 1780°, but the temperature at which the platinum lead becomes so weak that it is no longer able to resist the light stress which the springiness of the leads sets up; and this temperature might be materially below 1780°, and would indeed depend upon the stress under which the leads happened to be.

Plot your results with temperature as ordinate and time as abscissa. The results obtained with the thermo-electric pyrometer should fall in a very smooth curve. Repeat the heating and cooling three times, and calculate the maximum and average deviation from a perfectly smooth curve for each instrument.

Hand to the officer in charge your report, with a brief statement of your inference as to the relative merits of the three pyrometers, pointing out the specific advantages and disadvantages of each.

Experiment 16

THERMAL GRADIENT. — The purpose of this experiment is to show the considerable variations of temperature in a space which at first sight might seem uniformly heated.

Set the thermo-junction of a Le Chatelier pyrometer in the middle of the length of a glass combustion tube in a common combustion furnace, with the two leads extending in opposite directions, so that one lead passes out through a perforated clay or rubber stopper at the right-hand end, and the other lead through a like stopper at the left-hand end of the tube. The leads should be naked, i. e., not covered with any insulating clay tube as in the other experiments. Open the several gas cocks beneath the furnace wide, so as to supply the same quantity of heat throughout the length of the tube, and regulate the gas supply to the whole so that the thermo-junction may indicate a temperature between 475° and 500° C. After holding the temperature for 10 minutes between 475° and 500°, determine the temperature at other points in the length of the tube by drawing the thermo-junction along the tube, and record time, position and temperature. For a distance of 100 mm. at each end of the tube determine the temperature thus at points 15 mm. apart. Throughout the rest of the length determine it at points 25 mm. apart. Determine the temperature at the middle of the length of the tube after thus exploring the right-hand end, and again after exploring the left-hand end, i. e., at the middle and at the end of the series of tests.

From these determinations of the temperature at the middle of the tube, calculate the difference in temperature between the middle, as a datum point, and the various other points; and plot these results with horizontal distance as abscissa and temperature-

difference as ordinate. Scale for ordinates, 1 in. = 40° C.; for abscissæ, 1 in. = 2 in.

Note particularly the influence of the end cooling.

Experiment 17

THERMAL GRADIENT. — Repeat Experiment 16, making the following change. Having determined the thermal gradient as there directed, next make it as flat as practicable by regulating the supply of gas from the different gas-cocks, and find what degree of uniformity of temperature can thus be reached.

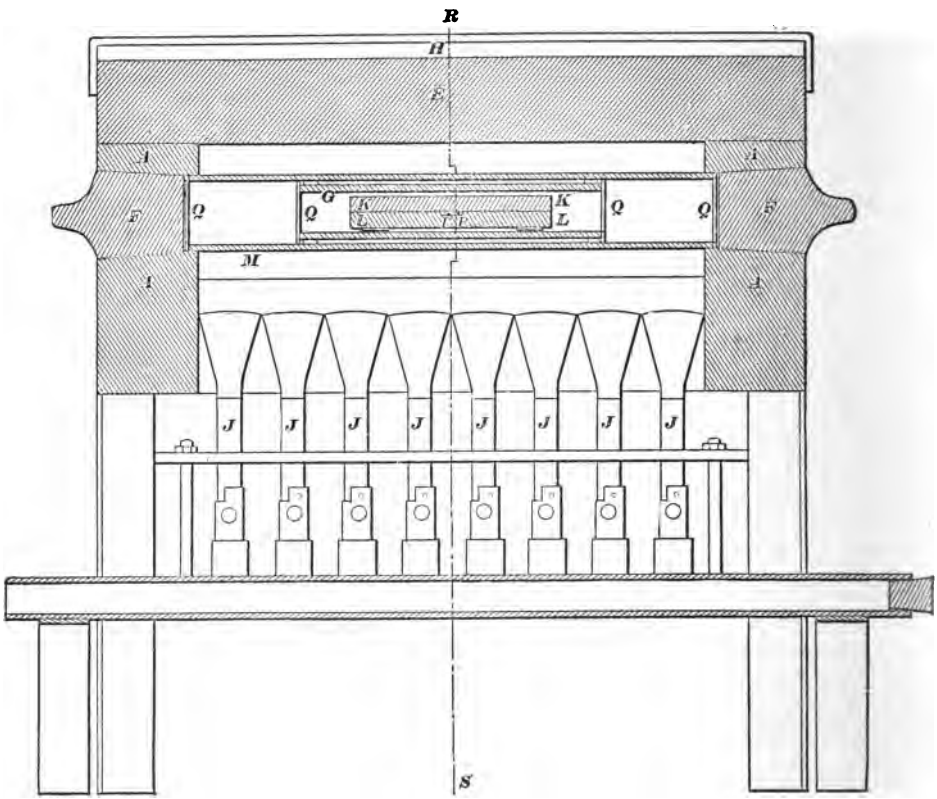


Fig. 15. — The Author's Double-Tube Muffle Gas-Furnace.
(Longitudinal Section.)

Legend:

F, clay-stoppers; *G*, copper tube or inner muffle; *J*, burners; *K*, bar to be heated; *L*, supporting bar of the same material as *K*; *M*, porcelain tube or outer muffle; *P*, wires leading to galvanometer; *Q*, diaphragm.

Scale 1 = 4.

Experiment 18

THERMAL GRADIENT. — Repeat Experiment 16 with the following variations:

Instead of a glass combustion tube use a porcelain tube* in the tubular muffle furnace shown in Fig. 15. Raise the temperature to about 800°, by opening wide the several gas-cocks under the tube, and regulating the supply of gas from the gas-main. Now determine and plot the thermal gradient as before.

Now try to make the temperature even throughout the length of the furnace by eye, partly opening or closing the several cocks under the tube so that the whole tube appears to the eye to be of the same color and hence temperature. Now determine the temperature at different points in the length of the tube as directed in Experiment 16, and plot as before. This will show how much the temperature may actually vary in a space in which to the eye the temperature appears to be quite uniform.

Experiment 19

ISOTHERMS. — The purpose of this experiment is like that of Experiment 16 (Thermal Gradient).

Cover the doorway of a gas-heated muffle furnace with a tile of such size that it leaves uncovered at the top of the doorway a space at least two square inches. Through a hole in the bottom of this tile pass the thermo-junction B (Fig. 16) of a Le Chatelier pyrometer, with insulating double-bored clay tubes C, lashed to an iron rod D, ¼ inch in diameter, and long enough to reach to the back of the muffle and serve as a handle for moving the couple about. Push the couple thus to point A, and hold it there until the gas supply has been so regulated that the temperature at A remains between 500° and 600° C. Now maintaining the gas supply constant, determine the temperature at A at intervals of 3 minutes for half an hour, so as to determine the rate at which its temperature is changing. Plot the temperatures

* A metallic tube of any kind would give trouble through short-circuiting the thermo-electric current.

Fig. 15 shows also a way of arranging steel bars for careful heating in this furnace; of course, these bars would not be put in the furnace for the present experiment, nor would the inner tube; so that the outer tube would be quite empty.

thus determined as ordinate and time as abscissa, and produce the curve thus obtained to cover a further period of 15 minutes. Now determine the temperature at different points on the floor of the muffle in the order here given: b, c, d, e, f, g, h, i, k, l, m, and so on: in other words, in the axis and at either side of the muffle every two inches between the back and front. Each set of three points such as cAb, fde, etc., lies on the circumference of a circle centred at E. Record the time at which each determination is made.

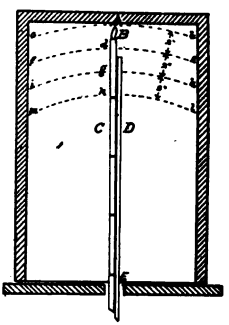


Fig. 16. — Positions of Thermo-Junction for Experiment 19.
Scale 1 = 16.

Legend: *A*, back wall of muffle.
B, thermo-junction.
C, double-bored clay tubes carrying leads of thermo-junction.
D, iron rod for moving thermo-junction.
E, entrance to muffle.
Small letters indicate the various positions of the thermo-junction.

Every ten minutes during the time covered by these observations make fresh determinations of the temperature of A, and by means of them correct the produced temperature curve of A. Now compare the temperature observed at each point with the temperature simultaneously existing at A as inferred from this produced temperature curve.

On a plan of the bottom of the muffle write the temperature differences between the several points and A as thus determined, and by means of these draw as contour lines the isotherms, or more strictly, lines of equal temperature-difference from A.

Submit your drawing to the officer in charge, with a written explanation of the chief variations in temperature detected.

Experiment 20

THE RELATIVE UNIFORMITY OF GAS AND SOLID, I. E., DIRECT-FIRED FURNACES. — Heat a coke-fired muffle furnace, with its doorway closed, to any convenient predetermined temperature (say 1200° C.), and record the temperature at a given point near its middle at intervals of 5 minutes for three hours, including the time of heating up from the cold.

Do the same for a gas-fired muffle, preferably of nearly the same size as the coke-fired one.

If practicable these two muffles should be near each other, so that this experiment can be carried out simultaneously in both, using either a pair of Le Chatelier pyrometers, or, better, a single pyrometer provided with a switch so that its galvanometer can be connected alternately with a thermo-junction in either furnace.

If two furnaces are thus simultaneously studied, two observers should coöperate; of these one should regulate the firing in both furnaces, trying to keep the temperature in each as constant as possible; the other should record the temperatures observed, and also the amount of time spent by his fellow in adjusting the temperature of each furnace separately.

REPORT, for each furnace separately,

(1) the length of time in reaching the predetermined temperature;

(2) the maximum deviation, and also the average deviation, from that predetermined temperature, starting from the time when that temperature is first reached;

(3) the number of minutes spent in regulating the temperature, including charging coke, removing ashes, adjusting valves, etc.

(4) Present a graphical representation, with temperature as ordinate and time as abscissa.

(5) State your inference as to the relative merits of these two systems of firing.

The student should appreciate that, in industrial metallurgy, the accuracy called for in most of the experiments in the present book is rarely to be had, and indeed rarely appropriate. This experiment may give him a foretaste, if that be needed, of the kind of irregularity which he will find in industrial operations,

an irregularity which could not be tolerated in that analytical study of the phenomena of metallurgy, which is the purpose of these experiments.

GROUP 3. — MELTING POINTS OF SILICATES, Etc.

Experiments 21 to 36

GENERAL DIRECTIONS CONCERNING DETERMINATIONS OF MELTING POINTS OF SILICATES AND OTHER EXPERIMENTS OF GROUP 3

Fineness. — The materials in each case are to be ground so that they pass a sieve of 100 meshes to the linear inch.

Quantity. — Prepare 200 grammes of each mixture.

Binding. — For binding material use common heavy lubricating oil in which 10 to 15 per cent of vaseline has been dissolved.

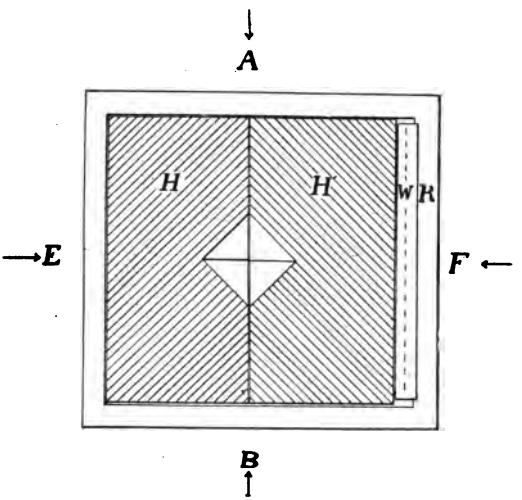


Fig. 17. — Section on *CD*.

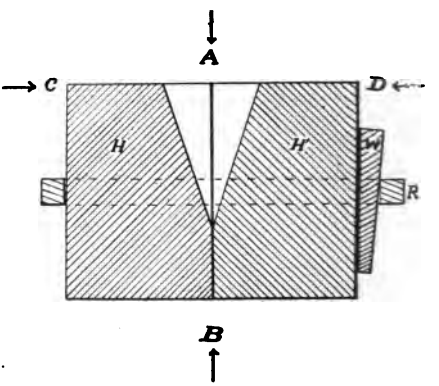


Fig. 18. — Section on *EF*.

Figs. 17 and 18. — Two Halves of Mould for Silicates, Assembled, Split along line *AB*. — Scale 1 = 2.

Legend: *H*, *H'*, the two halves of mould.
R, ring encircling mould.
W, wedge.

For dissolving the vaseline the oil should be heated. With this binding material even lime does not slack.

Ramming. — The materials of which the silicates are to be composed should be mixed with just enough of the binding

material to moisten them thoroughly. Now ram in a mould such as is shown in Figs. 17 and 18, a pyramid ¾ inch square and 1½ inches high, of each of the silicates. (If the arrangement shown in Figs. 21 to 23 is used, the pyramids should be truncated.) Ram the material in thin layers, scratching each layer before the next lot is added, so as to prevent cleavage surfaces. The material should be rammed very hard, using a rammer about ¼ inch in diameter.

After careful ramming, the pyramid should be removed from the mould, which should be opened by sliding its two halves past each other.

Drying. — The pyramids made in this way do not need the extremely slow drying which is usual when they are made with water. It is sufficient to set them in the doorway of a muffle furnace and raise them slowly to a dull red heat. The vaseline and oil will burn off, leaving the pyramids nearly white. They may then be put into the furnace in which the melting point is to be determined.

Precaution as to Lime. — The lime ought in every case to be freshly calcined, and all pyramids which contain lime ought to be tested immediately after preparing them, lest the lime should slack. In case of unavoidable delay, whether before or after firing, keep the pyramids in a dessicator to restrain slacking.

Determination of Melting Points. — Set a small muffle completely within a gas forge as shown in Figs. 19 and 20, closing the doorway of the muffle with a door in which two peep-holes are bored about 3 inches apart. Close the doorway of the gas forge with a fire-brick slab set on edge, bound with iron, and provided with a peep-hole. The peep-holes in both the inner and outer doors are to be kept closed with clay plugs, except when observations are to be made. To observe the condition of the pyramids, remove the outer door, and remove from the inner door one plug at a time, and for as short a time as practicable. Do not remove the inner door itself unless, thanks to the extreme evenness of temperature within the muffle, it becomes impossible to see the pyramids through the peep-holes. In that case remove the inner door, but only long enough to permit you to see the condition of the pyramids.

Put a graphite slab *b* on the floor of the muffle, and on it put the four pyramids as shown in Figs. 19 and 20.

Through the rear end of the muffle introduce the thermo-junction of a Le Chatelier pyrometer, boring through the back wall for this purpose as small a hole as will admit easily the double-bored clay tube carrying the leads of the thermo-junction. This should rest on the middle of the graphite slab, on which powdered chromite should be sprinkled, to prevent contact between the pyramids and the clay of this slab. The thermo-junction should lie in the centre of the muffle, and the four

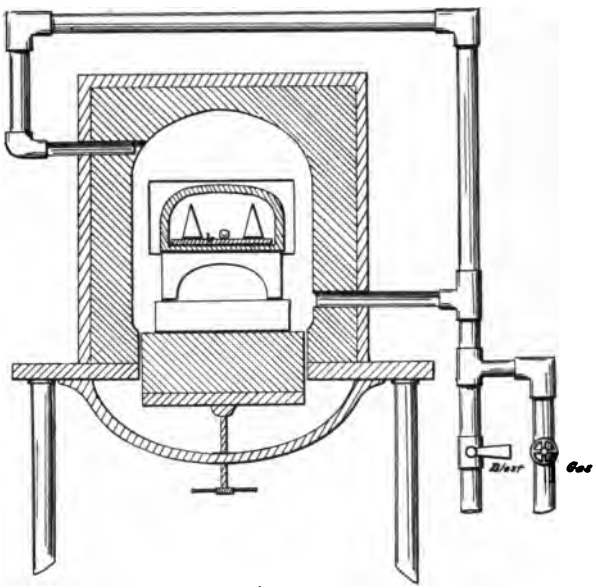


Fig. 19. — Gas Forge Showing Muffle Arrangement. (Cross-Section.)
Scale 1 = 8.

pyramids under observation should stand near it, 3 inches apart, and in line with the peep-holes in the muffle door.

Firing. — Raise the temperature very slowly, so slowly that at least half an hour is occupied in bringing the pyramids to visible redness. Then, turning on more gas and air, continue to raise the temperature gradually, so that at least an hour will be occupied in passing from visible redness to 1000°, and another hour in passing from 1000° to 1400°. Keep on raising the temperature until all the silicates have melted, but do not in

any case carry the temperature beyond 1600°, lest you injure the furnace. Record the temperature and the appearance of the four pyramids every ten minutes, and in particular the melting point of each pyramid.

Uniformity of Temperature. — Select six points in the region where the pyramids stand, readily reached by the thermo-junction; show their position by means of a sketch; determine the temperature at each of these points once for about every 100° rise of tem-

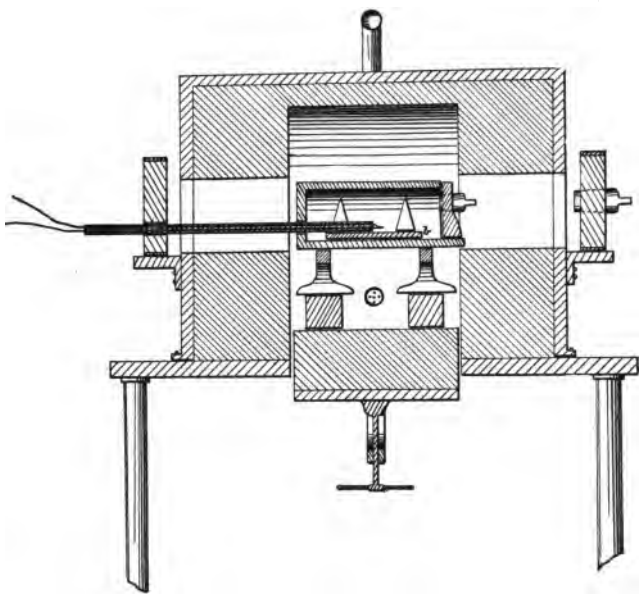


Fig. 20. — Gas Forge Showing Muffle Arrangement. (Longitudinal Section.) Scale 1 = 8.

perature; and include in your final report this sketch with a tabular statement (1) of these determinations, and (2) of the deviation at each point from the average temperature of the six points for each set of these determinations.

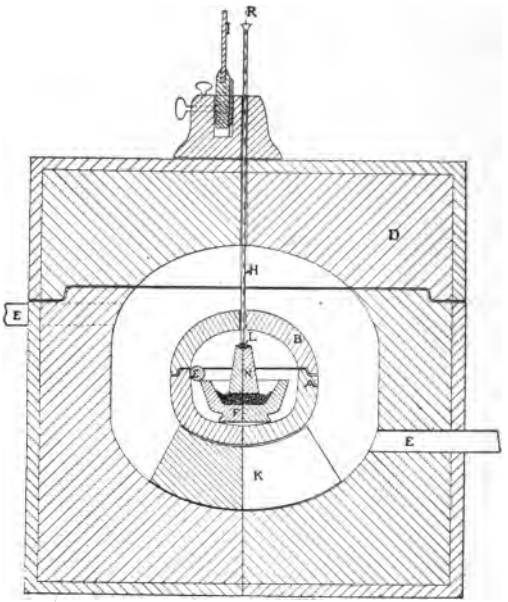
Report in tabular form the composition of the several pyramids and their melting points as thus determined, the manner of their melting, i. e., whether sudden or gradual, the greatest fluidity reached, their final condition, and also the matter specified in the paragraph preceding this one. Exhibit to an officer of the department.

Silicates

Special Muffle for Determining Melting Points of

Figs. 21 to 23 show a special form of muffle designed by the author for these experiments.

A is the muffle proper made of fire-clay, and closed at both ends.



Section UV S Section XY

Fig. 21. — Cross-Sections on UV and XY. — Scale 1 = 4.

B is the cover of the muffle.

C is the fire-clay outer wall of the furnace, and D the fire-clay roof.

E, E, ... are gas jets.

F is a scorifier, of which there are four in the muffle at a time, each holding one of the pyramids under test. The pyramids should be kept from actual contact with the scorifiers and tubes by means of partings of chromite or graphite.

G is the combustion chamber.

H is a porcelain indicator-tube resting on a chromite wafer on top of a pyramid, and passing up through the cover of the muffle and the roof of the furnace, so that its upper end is in plain view.

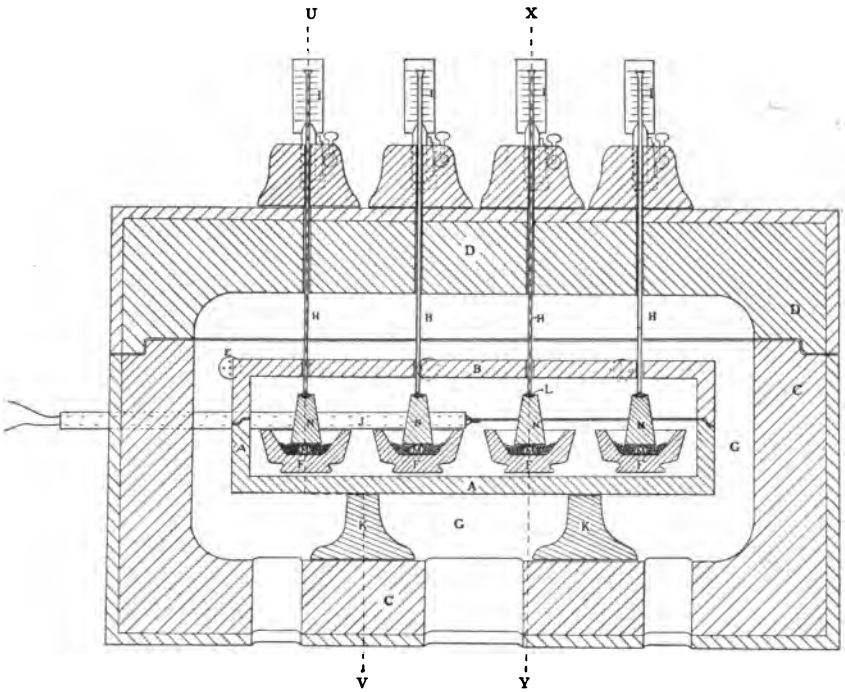


Fig. 22. — Longitudinal Section on RS. — Scale 1 = 4.

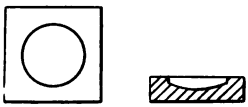


Fig. 23. — Chromite Wafer "L." — Scale 2 = 1.

Figs. 21 to 23. — Special Apparatus for Determining Melting Points of Silicates. (Designed by the Author.)

Legend:

A, muffle proper.
B, cover of muffle.
C, outer wall of furnace.
D, roof of furnace.
E, gas jet.
F, scorifier.
G, combustion chamber of furnace.
H, porcelain indicator-tube, resting on chromite wafer L.
I, scale for following the descent of the indicator-tube H.
J, double-bored clay tube, carrying leads of a Le Chatelier pyrometer.
K, shoe, supporting the muffle A.
L, chromite wafer, insulating the pyramid from the porcelain tube H.
M, chromite parting, insulating the pyramid from the scorifier F.
N, pyramid under test.

I is a scale for following the descent of the tube, and through it the progress of the melting of the pyramid on which this tube rests.

J is a double-bored clay tube carrying the leads of a Le Chatelier pyrometer.

K is a shoe on which the muffle rests.

L is a chromite wafer to insulate the pyramid from the porcelain indicator-tube.

M is a chromite parting preventing contact between the pyramid and the scorifier F.

N is the pyramid under test.

This muffle has not yet been made, and the design may need modification. It is so simple, however, and so likely to be useful without serious modification, that it may properly be described here.

Instead of the porcelain tubes, stout platinum wires may be used; but in this case they should be protected from contact with the fire-clay roof and muffle, for instance, by means of chromite plugs; or the roof and muffle may be made of magnesia, in which case they should be kept out of contact with fire-clay by a parting of chromite or graphite.

The essential features of the apparatus are the closed muffle, in which, because it is surrounded on all sides by the flame, the temperature should be very nearly uniform; the thermo-couple for following the temperature; and the porcelain tubes H for following the progress of the fusion. In all other arrangements which the author knows either the furnace cannot be opened until the end of the experiment, in which case our inspection tells us the effect of only the highest temperature reached, leaving us in doubt as to the temperature at which any pyramids which have melted actually did melt; or else the furnace is opened from time to time for inspection, in which case the very act of opening it cools its different parts unevenly, so that, until time has been given for the temperature to become even again, we cannot tell accurately what the temperature of the different parts is. Indeed, if the temperature rises only very slowly, as it should if it is to be very accurately even throughout the chamber, then that very evenness of temperature makes the pyramids invisible, not through any dazzling of the eye, but simply because they are of exactly the same tint as the enclosing walls, and hence cannot be distinguished from them by the eye.

The directions for firing with the arrangement shown in Figs. 19 and 20 apply, *mutatis mutandis*, to that shown in Figs. 21 to 23.

Experiment 21

SEGER PYRAMIDS.—Following the general directions, heat five Seger pyramids, Nos. 1, 5, 8, 9, and 10 to 1320° very slowly, so slowly that at least two hours will be occupied in rising from 1000° to 1320°. Record the temperature and the appearance of the pyramids every fifteen minutes.

The melting point, or rather the temperature assigned to each pyramid by the makers, is that at which it bends or "weeps" so far over that its apex touches the support on which it stands. Therefore note particularly this temperature for each of the pyramids which you test. Report the melting point of each of the five pyramids to an officer of the department.

REFERENCES: Seger, *Thonindustrie-Zeitung*, 1885, p. 121, and 1886, pp. 135 and 229. Le Chatelier and Boudouard, "Mesure des Températures élevées," 1900, p. 184. Hofman and Demond, *Trans. Amer. Inst. Mining Engineers*, XXIV, p. 42. The Author, *Engineering and Mining Journal*, June 7, 1890, p. 637.

Experiment 22

THE INFLUENCE OF FLUORINE ON THE FUSIBILITY OF EARTHY SILICATES (SINGULO AND ACID SILICATES OF LIME AND MAGNESIA, 2 CaO, MgO). — Following the general directions, determine the melting points of pyramids of each of the following compositions:

		O-ratio Base : Acid
(1)	$4CaO,2MgO,3SiO_2$	1 : 1
(2)	$10CaO,5MgO,18SiO_2$	1 : 2.4

(3) The same as (1), but intimately mixed with 5 per cent of fluorite.

(4) The same as (2), but intimately mixed with 5 per cent of fluorite.

In particular report (1) the difference in melting point of the two made without fluorite; (2) how many degrees the melting point of each silicate is lowered by the addition of 5 per cent of fluorite; and (3) what difference in the manner of melting, e. g., in its suddenness and in the resultant liquidity, is caused by the fluorite.

SLAG EXPERIMENTS NEEDING A VERY HIGH TEMPERATURE

Experiment 23

LIME-MAGNESIA SESQUI-SILICATES. — Following the general directions, determine the melting points of the following silicates:

	O-ratio Base : Acid
$4CaO,3SiO_2$	1 : 1.5
$4MgO,3SiO_2$	1 : 1.5
$2CaO,2MgO,3SiO_2$	1 : 1.5
$20CaO,8MgO,21SiO_2$	1 : 1.5

Experiment 24

LIME-MAGNESIA TRI-SILICATES. — Following the general directions, determine the melting points of the following silicates:

	O-ratio Base : Acid
$2CaO,3SiO_2$	1 : 3
$2MgO,3SiO_2$	1 : 3
$2CaO,2MgO,6SiO_2$	1 : 3
$20CaO,2MgO,33SiO_2$	1 : 3

Experiment 25

LIME-MAGNESIA 2.5 SILICATES. — Following the general directions, determine the melting points of the following silicates:

	O-ratio Base : Acid
$4CaO,5SiO_2$	1 : 2.5
$4MgO,5SiO_2$	1 : 2.5
$2CaO,2MgO,5SiO_2$	1 : 2.5
$16CaO,4MgO,25SiO_2$	1 : 2.5

Experiment 26

LIME SILICATES. — In the same way, following the general directions, prepare and test lime silicates with the oxygen ratio of base to silica as follows: 1 : 1, 1 : 2, 1 : 2.8, 1 : 3.5.

Experiment 27

CaO,MgO SILICATES. — In the same way, following the general directions, prepare and test silicates of lime and magnesia in the ratio of one equivalent of lime to one of magnesia, and with varying proportions of silica so as to bring the oxygen ratio of base to silica to 1 : 1, 1 : 2, 1 : 2.7 and 1 : 3.5.

Experiment 28

2 CaO,MgO SILICATES. — In the same way, following the general directions, prepare and test similar silicates but with two equivalents of lime to each equivalent of magnesia, and with the oxygen ratio of base to acid as follows: 1 : 1, 1 : 2, 1 : 2.4 and 1 : 3.5.

Experiment 29

3 CaO,MgO SILICATES. — In the same way, following the general directions, prepare and test silicates with three equivalents of lime to each equivalent of magnesia, and with the oxygen ratio of base to acid as follows: 1 : 1, 1 : 2, 1 : 2.4 and 1 : 3.5.

Prepare a diagram with melting point as ordinate and oxygen ratio of base to acid as abscissa, and draw on it four curves to represent the melting points of the four groups of silicates described in Experiments 26 to 29 inclusive.

Experiment 30

LIME-ALUMINA SILICATES. — In the same way, following the general directions, prepare four sets of lime-alumina silicates, respectively sub-silicates (oxygen ratio of base to acid 1 : 0.7), singulo-silicates, bi-silicates, and tri-silicates. In each of these prepare six silicates, or in all 24 silicates, with the oxygen ratio of the alumina to lime in each series as follows: 0 : 1, 0.1 : 1, 0.35 : 1, 0.65 : 1, and 1.2 : 1.

Prepare a diagram with melting point as ordinate and oxygen ratio of alumina to lime as abscissa, and on this diagram draw four curves, one for the sub-silicates, one for the singulo-silicates, one for the bi-silicates and one for the tri-silicates.

The results should show that, counting alumina as a base, the more acid the slag the less alumina is needed to give the greatest fusibility.

REFERENCES for experiments 22-30: Åkerman (Ledebur, "Handbuch der Eisenhüttenkunde," second edition, 1894, pp. 182-185). Also *Trans. Am. Inst. Mining Engineers,* XXVIII, p. 346.

INFERENCES FROM EXPERIMENTS 22 TO 29 INCLUSIVE. — From the results of Experiments 22 to 29 inclusive, select and tabulate (1) those tending to prove and (2) those tending to disprove each of the following propositions. Then, from a comparison of these condensed data, draw up and enunciate briefly your conclusions as to the degree of truth of each of these propositions.

A. The importance of the ratio of lime to magnesia in determining the fusibility is coördinate with that of the acidity.

B. Although the magnesia silicates are more infusible than the corresponding lime silicates, yet, in case of the singulo- and bi-silicates, replacing part of the lime by its atomic equivalent of magnesia (one of magnesia to three or even to two equivalents of lime), increases the fusibility of the silicate for given acidity. Hence the principle that a silicate of two bases is more fusible than the corresponding silicate of only one base.

C. If the magnesia be increased to the ratio of one equivalent to each equivalent of lime, the resultant silicate is not much more fusible, and may be less fusible, than the corresponding pure lime silicate.

D. In the case of the 3.5 silicates, replacing part of the lime by magnesia does not increase the fusibility.

E. The principle that a silicate of two bases is more fusible than the corresponding silicate of either of those bases alone, is of limited application.

Experiment 31

FORMATION POINT AND MELTING POINT. — It is generally believed that, in order to induce free silica and free bases mechanically mixed with it to combine, they have to be heated to a temperature considerably above the temperature at which the resultant silicate, once formed, will remelt; in short, that the formation point of a silicate is usually considerably above its melting point.

To illustrate this, prepare 200 grammes of a mixture of

Silica	41	per cent.
Alumina	25	"
Lime	34	"
	100	"

of such size that it will pass through a sieve of 20 meshes to the linear inch, but will not pass one of 40 meshes. Melt half of this in a brasqued crucible, and granulate it in water. Regrind this to the same size as before, and make from it three pyramids as described in the general directions for this group of experiments. Make also three like pyramids from the part of this silicate which has not been melted. On account of the coarse size of the particles, liquid asphalt should be used as a binding material instead of vaseline and oil.

Now determine the melting point of these pyramids, placing them side by side, as in the preceding experiments; in particular determine the difference between the melting points of the pyramids made from unfused and fused silicate respectively.

Hand to the officer in charge a statement of your inferences from this experiment, and your opinion as to the cogency of the evidence, together with your reasons.

Experiments 32 to 34

VERIFICATION OF RELATION OF FORMATION POINT TO MELTING POINT. — Experiment 31 may show that the formation point of a silicate is higher than the true melting point of the same silicate after it has actually formed. The question remains whether this difference is due simply to the imperfect mixing of the components of the silicate prior to its formation; and whether, given sufficient time, the separate components intimately mixed would or would not eventually unite and melt at the true melting point, i. e., whether, given sufficient time, the formation point would not be the same as the melting point. Experiments 32, 33 and 34 address this problem.

Experiment 32

VERIFICATION OF RELATION OF FORMATION POINT TO MELTING POINT. — Let Tm.p. = the melting point observed in Experiment 31.

Tf.p. = the formation point there observed.

Prepare a new pyramid of silica, lime, and alumina in the same proportions as in Experiment 31, and hold it for two hours as closely as possible at a temperature

$$T = \text{Tm.p.} + (\text{Tf.p.} - \text{Tm.p.}) \div 4,$$

in the electric-resistance crucible furnace. Record the temperature at intervals not greater than 10 minutes each, or if possible autographically. To prevent injury to the furnace the pyramid should be enclosed in a magnesia crucible, and should rest on a small plug of retort carbon to prevent contact with the crucible itself. Note carefully any signs of fusion or combination.

Experiment 33

VERIFICATION OF RELATION OF FORMATION POINT TO MELTING POINT. — Prepare another new pyramid, and hold it as nearly as you can at a temperature

$$T = \text{Tf.p.} - 25°$$

in the electric-resistance crucible furnace, with the same precautions as in Experiment 32, for two hours, unless it should melt before this. Throughout this time hold the temperature as nearly as you can at this point, but never allow it to rise above this point; in other words, the temperature is to be as little as possible below this point, but never above it. Record the temperature at intervals not greater than 10 minutes each, or preferably autographically.

From time to time note carefully whatever signs of fusion or combination there are.

Experiment 34

VERIFICATION OF RELATION OF FORMATION POINT TO MELTING POINT. — Repeat Experiment 31 with the following variations.

Preparation. The materials for each pyramid are to be ground to pass a sieve of 100 meshes to the linear inch, and are to be mixed very thoroughly by grinding in an agate mortar.

Heating. This is to be extremely slow, so slow that half an hour is occupied in the last 50° rise. Inspect the pyramids on reaching the Tm.p. found in Experiment 31, and, if either of them is then unmelted continue to raise the temperature at the same rate, and examine them after each 10° further rise.

For this experiment the electric-resistance crucible furnace must be used, with a differential rheostat. A strip of German silver serves as a simple and effective rheostat for this purpose.

From the results of Experiments 31, 32, 33 and 34 enunciate your inferences as to the relation between the formation point and the true melting point. In particular state whether the experiments which you have thus far tried are conclusive in your opinion; if not, what defect you find in them; and what farther experiments you suggest.

Experiments 35 and 36

THE FUSIBILITY OF LEAD SILICATES. — In these two experiments follow the general directions (p. 46), with the following exceptions.

(1) The materials must in each case pass a sieve of 60 meshes to the linear inch, and must be thoroughly mixed by grinding together in an agate mortar.

(2) No binding material is to be used, but the silicate is in each case simply to be formed in dry powder into a rough pyramid, at its natural angle of repose, in a 2½ inch scorifier.

(3) The weight of the mixture in each case is to be 30 grammes.

(4) The temperature need not be raised above 1350° C.

(5) At the end of the heating determine the fluidity roughly by dipping an iron rod into the molten silicate and withdrawing it.

Experiment 35

THE FUSIBILITY AS AFFECTED BY THE ACIDITY. — Determine in the way just described the melting points of the following mixtures:

		Litharge		Sand	
Lot	1	85	per cent	15	per cent
"	2	80	" "	20	" "
"	3	67	" "	33	" "
"	4	50	" "	50	" "

Report in tabular form the melting point of the several silicates, the manner of melting, i. e., whether sudden or gradual, and the greatest fluidity reached.

State concisely your inferences as to the influence of the percentage of silica on the melting point and the fluidity of lead silicates.

Experiment 36

INFLUENCE OF LIME AND FERRIC OXIDE ON THE FUSIBILITY AND FLUIDITY OF LEAD SILICATES. — Take five lots of a mixture of 85 per cent of litharge and 15 of pure silica sand (a mixture of which the melting point and fluidity has been determined in Experiment 35), and mix with them severally the following: —

With lot 1.....20 per cent of lime,
" " 2.....40 " " " "
" " 3.....20 " " " ferric oxide,
" " 4.....40 " " " " "

Determine as in Experiment 35 the melting point and the degree of fluidity of each, and report your results in tabular form.

State concisely your inferences as to the influence of lime and of ferric oxide on the fusibility and fluidity of this lead silicate, citing specifically the individual results on which each inference is based, and also any results which may oppose it.

GROUP 4. — PROPERTIES OF REFRACTORY MATERIALS

Experiment 37

CONTRACTION OF REFRACTORY MATERIALS. — Following the general directions for the experiments of group 3, prepare pyramids of the following substances:

(1) precipitated magnesium carbonate,

(2) magnesia made from the carbonate by gentle calcination, preferably at a temperature not above 800° C.,

(3) lime, calcined gently, preferably at a temperature not above 950° C.,

(4) raw dolomite,

(5) the mixture of lime and magnesia resulting from the gentle calcination of dolomite, preferably at a temperature not above 950° C.,

(6) fat clay,

(7) lean clay,

(8) burnt clay, in the shape of powdered clay fire-brick,

(9) silica sand.

The further treatment is to be like that set forth in the general directions for the experiments of group 3. After the

pyramids have been burnt white, cool and measure them accurately. Then heat them, four or five at a time, to 1200° C. in one of the forms of apparatus shown in Figs. 19 to 22.

If neither of these is available, then set the pyramids on a disc four inches in diameter, of retort carbon, or of graphite powdered with chromite to prevent contact between the clay of the graphite and the pyramids. Set the disc in a graphite crucible 5 inches in diameter, and heat this in a crucible furnace for 30 minutes to between 1150° and 1250° C. If a gas crucible furnace is used, a close enough measurement of the temperature can be had by placing the thermo-junction alongside of the crucible, and raising the temperature so slowly that 30 minutes are occupied by the last 50° rise. If a coke-fired crucible furnace is used, the thermo-junction should be set within the crucible itself.

Cool, remeasure the several pyramids, and calculate their cubic contraction. Report this in tabular form, first in percentage of the initial volume, and then in percentage of the contraction undergone by the magnesia.

Experiment 38

CONTRACTION OF REFRACTORY MATERIALS. — Repeat Experiment 37 with a new set of pyramids, but this time carry the temperature to 1300°, and again cool and remeasure. Then reheat this same set to 1500°, and then again to 1700°, remeasuring after each heating. Extend the table which gives the results of Experiment 37 so as to include those of Experiment 38.

These reheatings to 1500° and 1700° should not be made in a muffle, unless by advanced and very cautious students, because of the danger to the outer furnace which the attempt to reach such a temperature inside a muffle implies. To reach these temperatures, use either an open crucible as described in Experiment 37, or better a gas forge like that of Figs. 19 and 20 with the muffle omitted. In this case set a piece of fire-brick, about 6 in. x 4 in. x 1 in., on edge to windward of the pyramids, to protect them from the direct impact of the flame.

This experiment will show that magnesia not only contracts greatly when heated, but that, no matter how highly it has been heated, it contracts still more if reheated to a still higher temperature. This is true of dolomite, but to a much smaller degree,

while lime is not greatly affected by such higher reheating. This behavior of magnesia and dolomite calls for special precautions. For instance, when these substances are to be used for furnace linings they should first be raised to a temperature at least as high as any to which the lining is to be exposed in use, so that they may before use undergo the shrinkage corresponding to that temperature, lest the lining itself should contract in use, and thus crack.

Clay behaves like magnesia, contracting afresh with each exposure to a new temperature higher than any to which it has previously been exposed, the principle on which Wedgwood's pyrometer was based. In using clay fire-bricks this contraction is not in general to be considered, simply because the bricks themselves have already been heated in the brick-kiln to so high a temperature. But in using raw clay the metallurgist has to guard carefully against this great contraction, by mixing with his clay a great excess of some other substance which does not thus contract on heating, such as quartz sand, ground fire-brick, etc. In general, while he uses raw clay extensively, he uses only enough of it in any given refractory mixture to give the plasticity and cohesion needed.

Experiment 39

SLACKING OF BASIC REFRACTORY MATERIALS. — Expose the pyramids resulting from Experiments 37 and 38 to the air, and watch their behavior at intervals for a month, recording their appearance once a day for a week, and thereafter once a week. It will be found that magnesia does not slack at all; that the dolomite slacks but slowly, and that which has been most highly heated slacks less than that which has been less strongly heated; but that the lime slacks very rapidly.

Experiments 40 to 45

CORROSION OF REFRACTORY MATERIALS BY SILICATES. — This series of experiments investigates the corrosive action of different silicates upon some of the principal kinds of refractory materials with which industrial furnaces are lined. To this end six silicates of widely different composition are prepared, one for each of these six experiments; and in each experiment one of these

silicates is heated or melted in contact with four different refractory materials, viz.: — magnesia, chromite, silica (in the form of Hessian crucibles) and fire-clay.

The experiment upon chromite may be made with clay crucibles lined with chromite, as described in the appendix. For the experiments upon the three other materials the commercial magnesia, Hessian, and clay crucibles may be used, but care must be taken that the magnesia crucibles are pure. (See appendix.) If the commercial magnesia crucibles cannot be had, then a magnesia lining may be rammed inside a chromite lining, as described in the appendix.

Preparation of Silicates. — Of each of the silicates prepare 1 kg. Those free from oxide of iron may be prepared by melting their constituents, previously finely ground and thoroughly mixed, in a brasqued crucible, and granulating the molten silicate, by pouring it into water.

This is recommended as an easy way of insuring that the different lots of any given silicate, both in the original experiments as here set forth and in any repetitions or modifications of them, shall be substantially identical in composition.

The silicates containing iron oxide cannot be thus prepared in bulk, because the carbon of the brasquing would be likely to reduce part of their iron to the metallic state. We have therefore to rely on the usual precautions to insure constancy of composition. In particular, the constituents should be ground so as to pass through a sieve of 80 meshes to the linear inch, and very thoroughly mixed. A convenient way is to mix with a pure rich heating-furnace cinder made on a basic bottom, or with puddling cinder, enough silica or lime to give approximately the composition directed.

It is not necessary that the composition should be accurately that here prescribed; a rough approximation will in general suffice. It is understood that the iron silicates will in melting pass more or less to higher oxides. This, however, need not interfere seriously with the experiment.

General Procedure. — The four crucibles to be used in a given experiment, after being filled with their charge of silicate, are to be set in a four-holed graphite slab A, Figs. 24 and 25, which in turn rests upon a short piece D cut from the lower part of a graphite crucible about 12 inches in diameter. In the bottom

of this are placed two layers of material obtained by breaking up the thoroughly clean parts of old graphite crucibles. The upper layer C is in coarse fragments, and its object is to soak up any

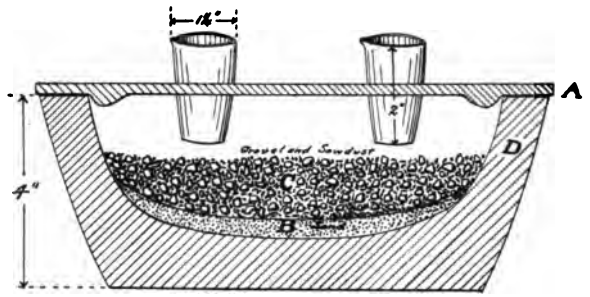


Fig. 24. — Section on *XY*.

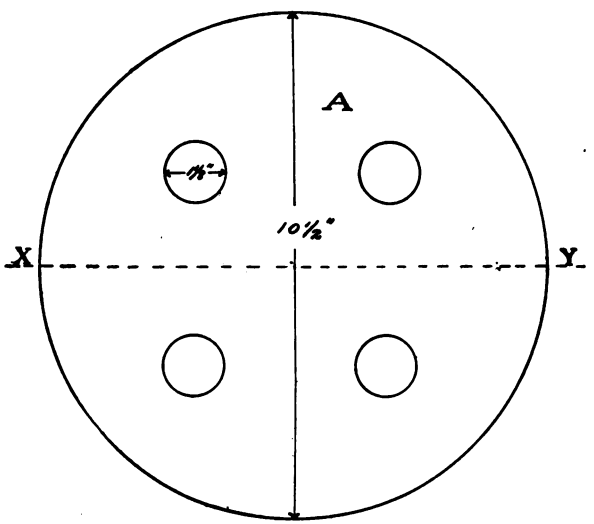


Fig. 25. — Plan of Graphite Slab, *A*.

Figs. 24 and 25. — Arrangement of Crucibles for Experiments on Corrosion of Refractory Materials. — Scale 1 = 4.

Legend: *A*, graphite slab supporting the four crucibles under test.
B, arresting layer of sand.
C, absorbing layer of gravel and sawdust.
D, stump of a graphite crucible.

molten silicate which may bore through any of the small crucibles under test, lest it attack and bore through the graphite crucible itself, and in turn injure the furnace bottom. The lower layer B is ground to pass a sieve of 20 meshes to the linear inch, and its object is to prevent any of this molten silicate from reaching the bottom of D. In short, C is a sponge and B an arresting layer.

Silica pebbles and sand may be used for this purpose instead of fragments of graphite crucibles; in this case the pebbles should be mixed with sawdust, lest their expansion burst the crucible D.

The crucibles thus assembled are to be heated in a gas crucible furnace, the temperature of which is to be controlled by a Le Chatelier pyrometer. The thermo-junction of this is to be set as near as practicable to the assembled crucibles. As exact temperature measurements are not needed, it is not essential that the thermo-junction should have accurately the temperature of the crucibles themselves; nevertheless the temperature should thus be roughly controlled, lest it either fail to rise to the melting point of the silicate used, or rise so high as to injure the furnace. With all undergraduate students, even after they have had considerable experience, this should be insisted upon. A gas crucible furnace suitable for this purpose is shown in Fig. 26.

Raise the temperature slowly, so that at least 30 minutes are occupied for every 50° rise from 1200° upward; and observe the condition of the charges in the four crucibles at short intervals, preferably of 15 minutes.

If the molten silicate sinks down deep in the crucible, this may be due either to its having corroded the crucible so as to bore a hole through it and run out; or to its having been soaked up by the walls of the crucible. To determine which of these has happened, pour into the crucible without removing it from the furnace a second, and if necessary to convince yourself, then a third crucibleful of the same silicate.

After the silicate has melted in all the crucibles the temperature should be prevented as far as possible from further rise; in other words, after fusion the temperature should be held above, but as little as possible above, the melting point.

In case none of the silicates sink down deep, so that a second lot of silicate is not added, then the crucibles should be withdrawn for examination 20 minutes after complete fusion, or as near to this time as practicable.

In case one or more of the silicates have sunk deep, so that a second or a third charge is added to determine the cause of this sinking, then the crucibles should be withdrawn as soon as the second charge (or the third if there are three), has melted.

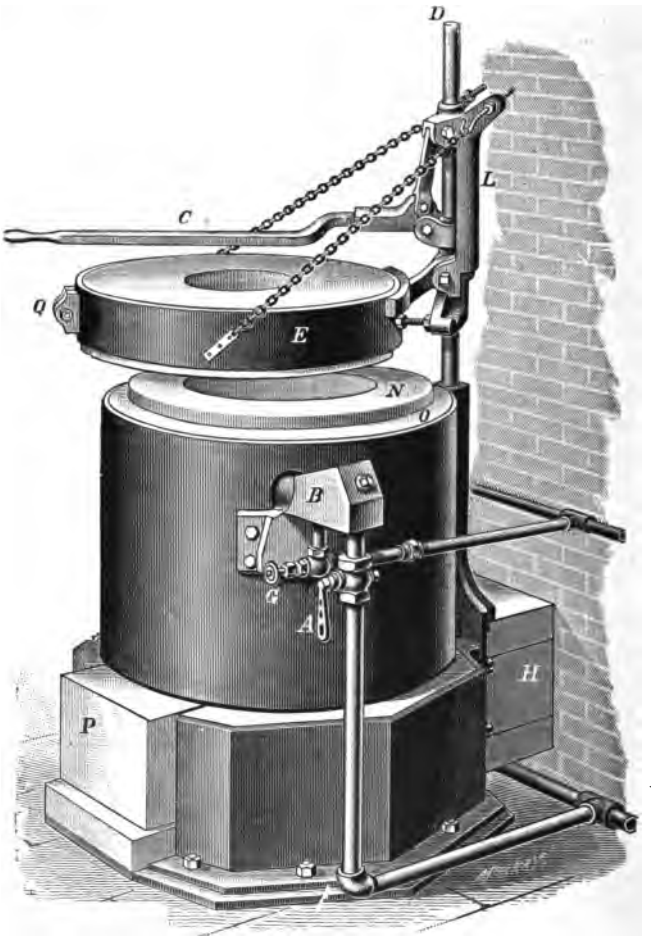


Fig. 26. — Crucible Furnace with Down Draft. (American Gas Furnace Co.)

Legend: *A*, air valve.
B, burner.
C, lever for raising cover.
D, rod carrying cover-lifting mechanism.
E, cover.
G, gas valve.
H, draft flue.
L, cover lift.
N, lining.
O, backing.
P, plug.
Q, clamp.

It is better, when practicable, to draw each crucible separately and empty it; for this gives the best opportunity to see, after it has cooled, how much it has been corroded. If this be impracticable, then draw all the crucibles together, and examine them, first by inspection from without both while hot and after cooling, and then by breaking them up and examining their walls. Information as to whether a given silicate has corroded a given crucible so badly as to run through its walls can often be had by looking for stalagmites of silicate on the surface of layer C.

It will sometimes happen that a silicate may run out of a crucible, not through corroding its walls, but through a crack or other mechanical defect in the crucible; and mere examination of the emptied crucible does not always tell clearly to what cause the escape of the silicate is due. Whenever there is reason to suspect that this escape is due to a mechanical defect, the experiment should be repeated.

A special reason why the crucibles should be examined before complete cooling is that some silicates, especially those rich in lime, disintegrate with great expansion during or immediately after solidification; and this may both burst the crucible, and mislead you into supposing that the silicate has not melted at all.

Experiment 40

Corrosion of Refractory Materials by Ferrous Singulo-Silicate ($2\,FeO,SiO_2$). — Determine the action of molten ferrous singulo-silicate, $2\,FeO,SiO_2$, ferrous oxide 71 per cent, silica 29 per cent, on silica, fire-clay, magnesia and chromite, in the manner just set forth.

Experiment 41

Corrosion of Refractory Materials by Ferrous Bi-Silicate (FeO,SiO_2). — The same for ferrous bi-silicate, FeO,SiO_2, ferrous oxide 55 per cent, silica 45 per cent.

Experiment 42

Corrosion of Refractory Materials by an Earthy Singulo-Silicate. — The same for the earthy singulo-silicate containing

Silica	42	per cent
Alumina	24	" "
Lime	21	" "
Magnesia	13	" "
	100	" "

Experiment 43

CORROSION OF REFRACTORY MATERIALS BY AN ACID (2.5) EARTHY SILICATE ($16CaO,4MgO,25SiO_2$). — The same for the acid earthy 2.5 silicate (i. e., with the oxygen ratio of base to acid 1 : 2.5), containing four equivalents of lime to one of magnesia, $16CaO,4MgO,25SiO_2$.

Experiment 44

CORROSION OF REFRACTORY MATERIALS BY AN ACID LIME-FERROUS OXIDE SILICATE. — The same for the acid silicate of lime and ferrous oxide, containing

Silica	52	per cent
Lime	26	" "
Ferrous oxide	22	" "
	100	" "

Experiment 45

CORROSION OF REFRACTORY MATERIALS BY A BASIC LIME-FERROUS OXIDE SILICATE. — The same for the basic silicate of lime and ferrous oxide, containing

Silica	32	per cent
Lime	48	" "
Ferrous oxide	20	" "
	100	" "

Results and Inferences. — Present your results in a table with a column 2½ inches wide for each of the four refractory materials, and with lines 2 inches apart, one for each silicate. In each of the divisions thus made record the effect of the different silicates upon the different refractory materials.

State clearly but briefly your inferences from your results as to each of the following propositions, citing each result which

supports and also each which opposes each proposition. This statement is to be in addition to the table just referred to.

(1) Ferruginous silicates, even when acid, corrode sand and clay crucibles, and when basic they corrode them very actively.

(2) For given acidity, ferruginous silicates are more corrosive toward all these refractory materials than earthy silicates.

(3) Neither the acid earthy, nor the basic earthy silicates corrode any of these refractory materials rapidly, if free from iron oxide.

(4) For given acidity, silicates containing both iron oxide and earthy bases are intermediate in corrosiveness between the pure iron and the pure earthy silicates.

(5) For given ratio of iron oxide to earthy base, acid silicates corrode basic refractory materials most, and basic ones corrode acid materials most.

(6) Chromite is less easily corroded than silica, fire-clay or magnesia by molten silicates in general, in the sense that it excels these other materials greatly in the range of composition of the molten silicates which it withstands without being seriously corroded.

GROUP 5. — IRON AND STEEL

Experiment 46

THE DESULPHURIZATION OF IRON BY MANGANESE. — Manganese desulphurizes molten cast iron, apparently by causing the gradual liquation of manganese sulphide, which rises to the surface, where its sulphur is oxidized by the air.

Melt in a covered graphite crucible under a layer of powdered charcoal 10 pounds of cast iron containing at least 1 per cent of sulphur, and not over 0.25 per cent of manganese. Holding it molten take out a 200-gramme sample at the end of thirty minutes by means of a clay-washed iron spoon. Now add enough ferro manganese or spiegeleisen to introduce 2 per cent of manganese, and take out a sample in five minutes, and four others at 15-minute intervals. Determine the manganese and sulphur in the whole series of samples.

This will show the progress of the removal of sulphur, and the accompanying loss of manganese.

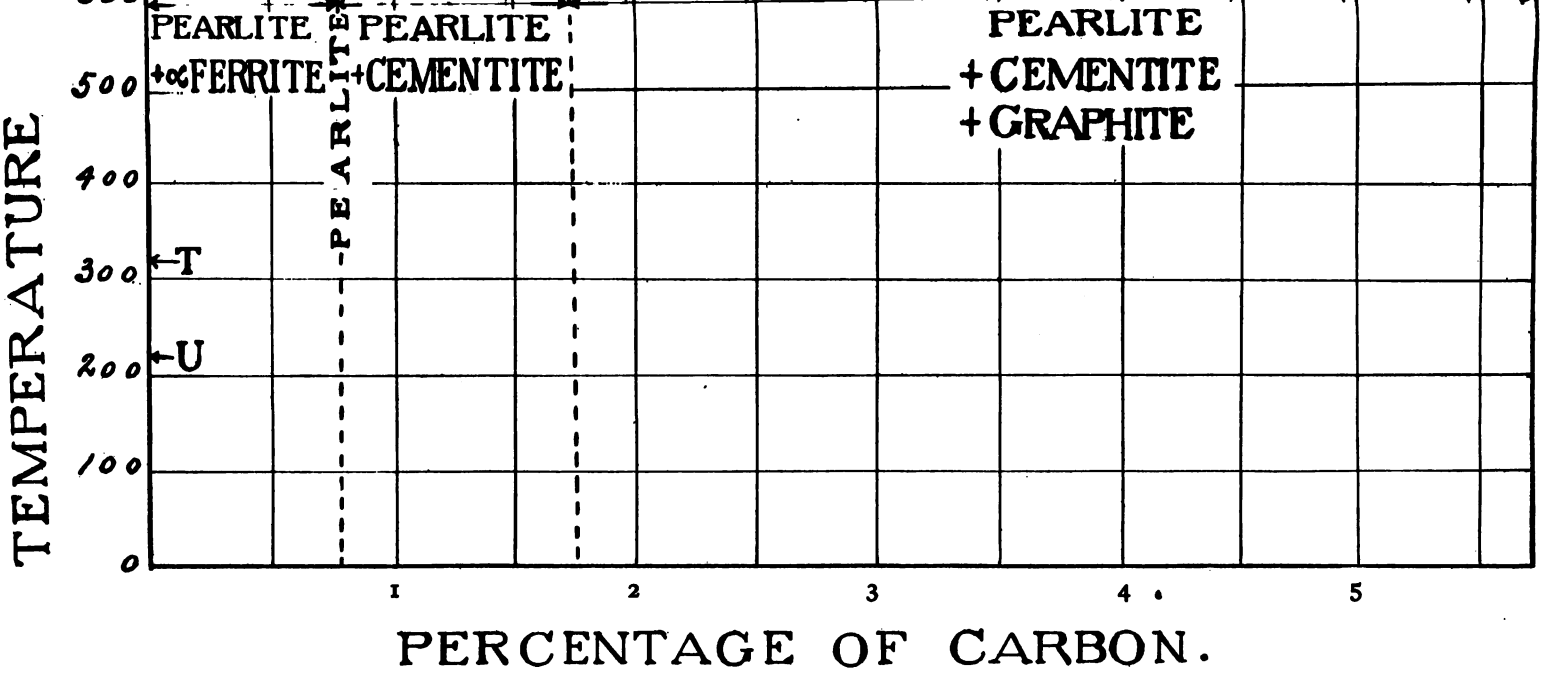


Fig. 27. — Roberts-Austen's Diagram, with certain of Roozeboom's Modifications.

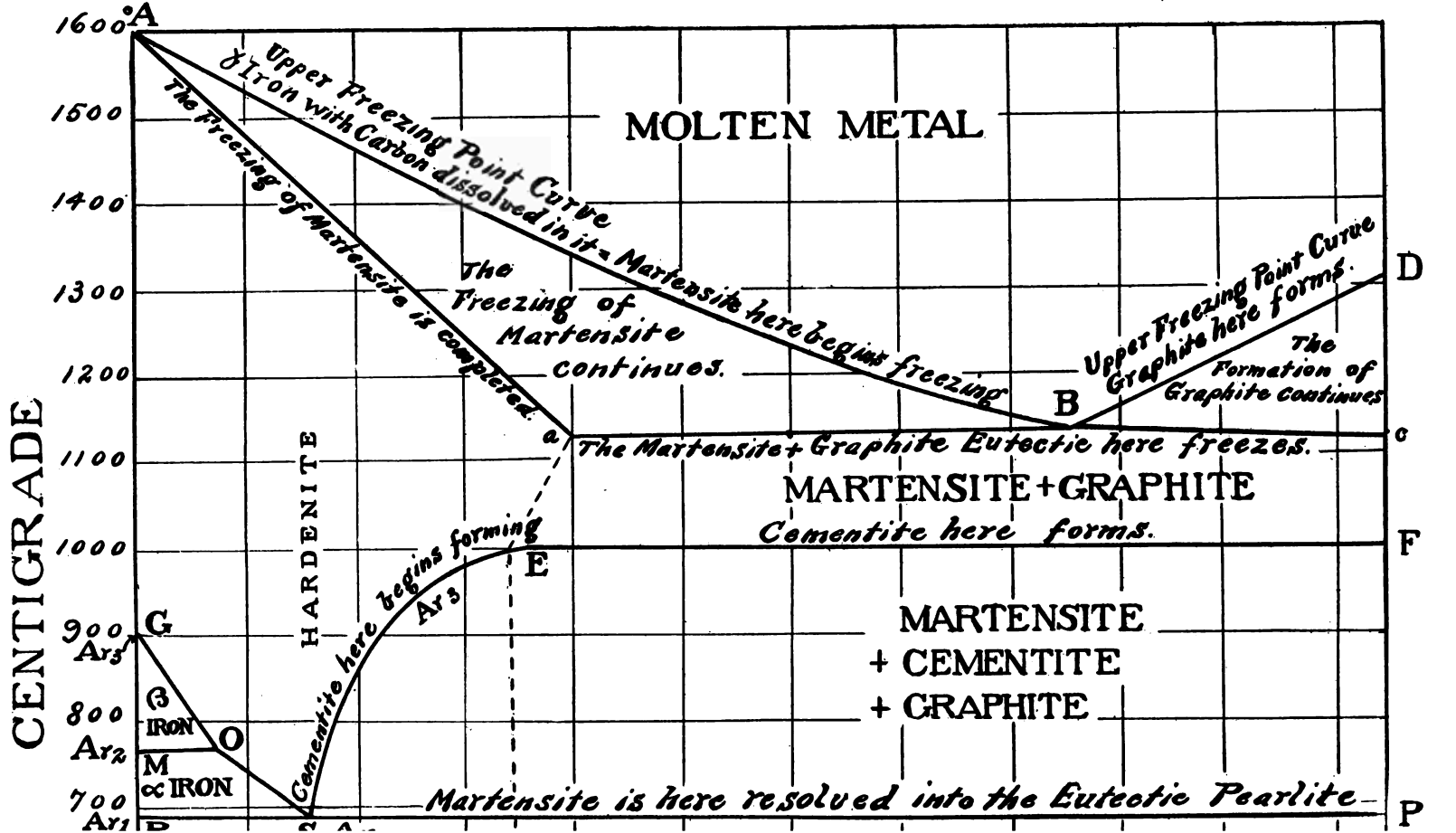
Note. — The ROMAN CAPITAL LETTERS indicate the constituents normal and stable, or at least persistently present, in each of the fields into which the diagram is divided. The transformations which in cooling occur at the horizontal and oblique boundary lines of these fields are indicated in small letters.

The dotted lines are boundaries, due to variations in composition, of the regions of the several sets of constituents, e.g., pearlite + α ferrite, etc.

T U = tempering range.

References: Roberts-Austen, Fifth Report Alloys Research Committee.

Roozeboom, *Zeitschrift für physikalische Chemie*, XXXIV, 1900, p. 437; *The Metallographist*, III, p 293; "Le Fer et l'Acier au Point de vue de la Doctrine des Phases," "Contribution à l'Étude des Alliages," Paris, 1901 (translation by F. Osmond), p. 327.


CENTIGRADE
1600°
1500
1400
1300
1200
1100
1000
900
800
700
A
B
C
D
E
F
G
P
MOLTEN METAL
γ Iron with Carbon dissolved in it = Martensite
Upper Freezing Point Curve
The Freezing of Martensite is completed
The Freezing of Martensite continues.
Upper Freezing Point Curve
Graphite here forms.
The Formation of Graphite continues
a
The Martensite + Graphite Eutectic here freezes.
MARTENSITE + GRAPHITE
Cementite here forms.
HARDENITE
Cementite here begins forming
Ar 3
MARTENSITE
+ CEMENTITE
+ GRAPHITE
Ar3
Ar2
Ar1
β IRON
M
α IRON
O
Martensite is here resolved into the Eutectic Pearlite

Throughout the experiment add enough charcoal in lumps about one inch cube to keep from one to four lumps upon the surface of the molten iron. This is to prevent the iron from decarburizing, and thus becoming so infusible as to solidify.

Skim off the slag at short intervals.

Hand to the officer in charge your results in tabular form, with a brief statement of the law representing the relation between (1) the degree of desulphurization, (2) the removal of manganese, and (3) time, i. e., the length of the desulphurizing period.

Experiment 47

VERIFICATION OF ROBERTS-AUSTEN'S DIAGRAM. — Determine the heating and cooling curves of six lots of steel containing approximately the following percentages of carbon: 0.20, 0.40, 0.60, 0.80, 1.25, 1.50 and 2 per cent. The steel should be as nearly free from elements other than iron and carbon as possible, and should be in wads as shown in Fig. 13. The heating and cooling may conveniently be done in the electric-resistance crucible furnace, Fig. 13.

The galvanometer used for this experiment should give large deflections, and the heating and cooling should be very slow, so that the time error may be small. The cooling should be retarded purposely. For instance, if the furnace which contains the steel is a gas-fired one, then in cooling the gas should simply be turned down somewhat, instead of being cut off wholly. If it be an electric one, the current should be reduced, not stopped.

To determine the upper critical points, those above the recalescence, great care is needed, and two observers should work together: or, better, an autographic galvanometer should be used.

To make your results comparable, the rate of cooling should be as nearly as possible the same for the different kinds of steel, and so should the rate of heating.

Plot your results with temperature as ordinate and percentage of carbon as abscissa, drawing curves through the retardation points both in heating and in cooling. Each retardation usually covers a considerable range of temperature, and that temperature should be selected for plotting at which this retardation is most pronounced.

Report any discrepancies between your results and Roberts-Austen's diagram, with your explanation.

Experiment 48

MANGANESE STEEL OF 12 TO 14 PER CENT OF MANGANESE. — Heat six bars of manganese steel, approximately $^5/_{16}$ inch square, and 9 inches long, in a muffle furnace or otherwise, to approximately 1300° C. Cool one of them in lime, one of them in air, one by means of a blast of air, one by quenching it in linseed oil, one in boiling water, and one in iced brine. Determine the ductility of these by bending in the drop-test machine (Fig. 8) and measuring the degree bent on rupture.

Determine the hardness with the sclerometer, and qualitatively with a file.

The results should show that, while the hardness is not appreciably affected by the rate of cooling, so that the metal is always so hard that it cannot be filed, yet the more rapidly it is cooled the more ductile does it become.

REFERENCES: Hadfield, (*Jour. Iron and Steel Inst.*, 1888, II; *Proc. Inst. Civ. Eng.*, XCIII, III, 1888; U. S. Patents 303, 150-1; British Patents 200 of 1883, and 8268 and 16,049 of 1884). Howe, "Metallurgy of Steel," p. 361; *Trans. Am. Inst. Min. Eng.*, XXIII, pp. 466-476.

Experiment 49

INFLUENCE OF RATE OF COOLING UPON THE FRACTURE OF CAST IRON.* — Melt in a covered and well-luted graphite crucible

* Chilled cast-iron railroad car-wheels are a convenient source of cast-iron suitable for this experiment. After the car-wheel has been broken by means of a drop or of a heavy sledge to fragments as small as practicable, these may in turn be further broken up by heating them to redness, quenching them in water, and then again sledging them. The heating and quenching in water is for the purpose of cracking the cast-iron, so that it will break the more readily. It is important that the pieces of cast-iron should be small, so that when melted in this experiment they may be completely covered by the charcoal, and may remain covered. If they are exposed to the air in melting down, both their silicon and carbon will oxidize. Now, in order that the experiment should succeed, the cast-iron must be rich in carbon, and the silicon should be between the limits 0.50 and 0.90 per cent. If there is more than 0.90 per cent silicon, then even the bar cast in the cold iron mould may be gray; whereas if the cast-iron contains less than 0.50 per cent silicon, then even that cast in the brick mould may be white. In short, cast-iron of the composition above prescribed will show very distinctly the effects of the rate of cooling, but if the composition be outside of these limits these effects may not be prominent. If the silicon is too low it may be raised by a slight addition of carborundum.

under a 2-inch layer of powdered charcoal, 10 pounds of cast iron containing not over

0.10 per cent sulphur,
0.20 " phosphorus,
0.50 " manganese,

and between 0.50 and 0.90 per cent. silicon.

As the charcoal burns away, replace it with lump charcoal, to prevent the iron from becoming decarburized.

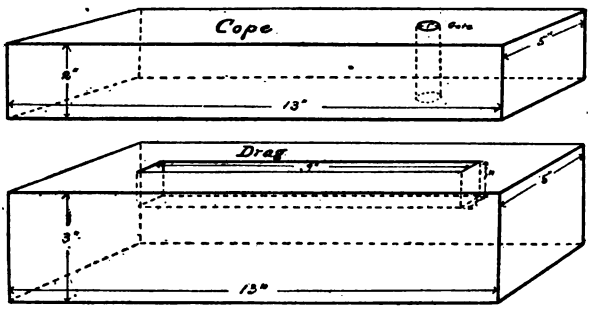
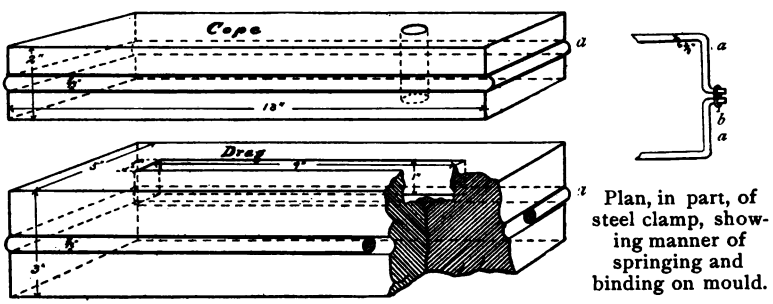


Fig. 28. — Cast Iron Ingot Mould.



Plan, in part, of steel clamp, showing manner of springing and binding on mould.

Fig. 29. — Fire-Clay Ingot Mould.
a, steel clamp. *b*, ring binding clamp in place.

When the iron is thoroughly melted pour two ingots, each 1 inch square and 9 inches long, one in a cold iron mould (Fig. 28), and the other in a pre-heated brick mould (Fig. 29). This with its cover may for convenience be pre-heated on the crucible furnace during the melting of the iron.

The ingot quickly cooled by casting in the iron mould will be hard, brittle, and white in fracture; that cooled slowly by casting in the pre-heated brick mould will be gray in fracture, soft, and relatively ductile, i. e., ductile for cast iron.

Test both bars in the drop-testing machine (Fig. 8), but instead of dropping the ram from the whole height, drop it first from a height of 1 inch, then from a height of 2 inches, and so on, increasing the fall by 1 inch each time, until the bar breaks. Compare the fall required to break the two bars.

Determine the hardness of both bars with the sclerometer, polishing on each a space 1 inch square for this purpose.

Experiments 50 to 55

BRINELL'S TESTS FOR TOOL STEEL. — Let the student first read the following notes, and then perform the experiments graphically shown in Figs. 30 to 35. Let him then assemble his fractures, label them distinctly, compare them with the framed series of Brinell fractures, and hand them to the proper officer. While these experiments are the better for being carried out accurately with a pyrometer, yet even without such accuracy they give very valuable information, especially about the thermal treatment of tool steel. They therefore commend themselves particularly to those who have no accurate means of measuring the temperature.

EFFECT OF HEAT-TREATMENT ON FRACTURE. — *Graphical Representation of Brinell's Results. — Steel of 0.75 per cent of Carbon.* — Figs. 30 to 35 represent the conditions and results of experiments on steel, containing 0.75 per cent of carbon. Each line beginning with O and ending with V represents one experiment. In each case the metal is gradually heated to a certain temperature, indicated by the point at which the line doubles and begins to descend. In most cases the temperature now descends without interruption: but in the last test the cooling is interrupted, as indicated by a second doubling and the re-ascent of the line.

There are two kinds of fractures: main and transition. There are four main fractures:

C — unhardened, fine crystalline, characteristic of well-treated unhardened steel.

A — unhardened, coarse crystalline, characteristic of overheated unhardened steel.

Graphical Representation of Brinell's Tests

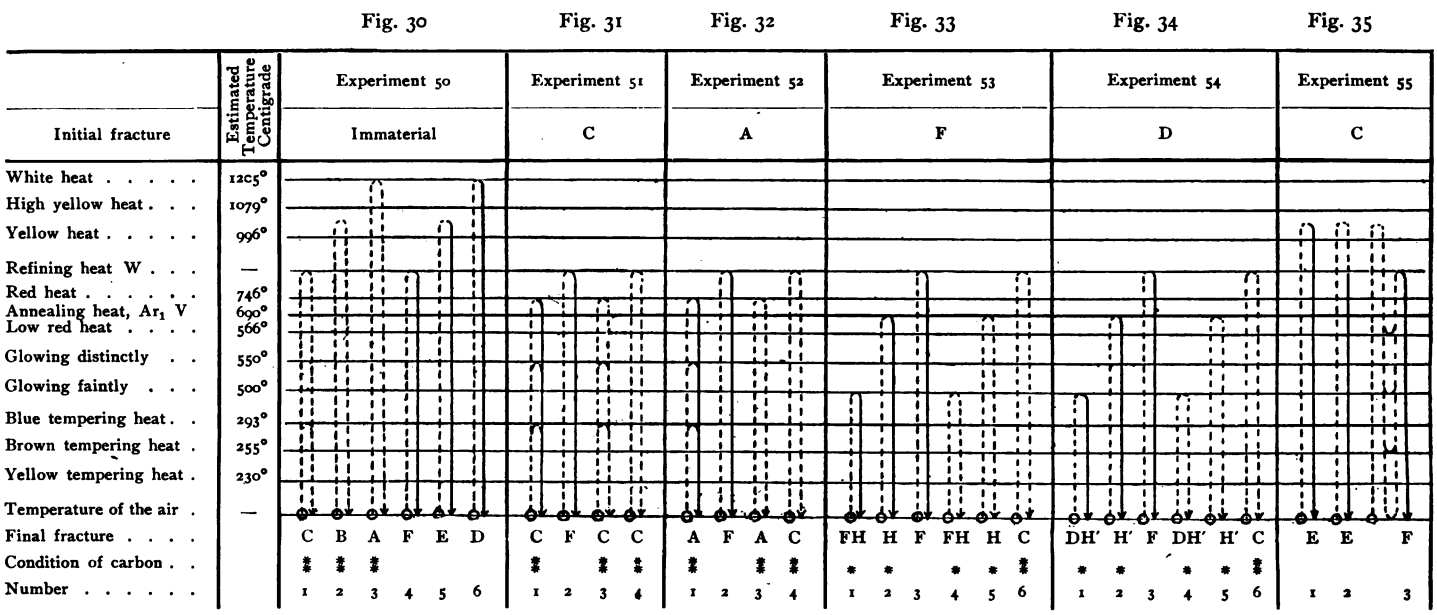


Fig. 30
Fig. 31
Fig. 32
Fig. 33
Fig. 34
Fig. 35
Initial fracture
Estimated Temperature Centigrade
Experiment 50
Experiment 51
Experiment 52
Experiment 53
Experiment 54
Experiment 55
Immaterial
C
A
F
D
C
White heat . . . . . 1205°
High yellow heat . . . 1079°
Yellow heat . . . . . 996°
Refining heat W . . . —
Red heat . . . . . 746°
Annealing heat, Ar1 V 690°
Low red heat . . . . 566°
Glowing distinctly . . 550°
Glowing faintly . . . 500°
Blue tempering heat . . 293°
Brown tempering heat . 255°
Yellow tempering heat . 230°
Temperature of the air . —
Final fracture . . . . C B A F E D | C F C C | A F A C | FH H F FH H C | DH' H' F DH' H' C | E E F
Condition of carbon . .
Number . . . . . 1 2 3 4 5 6 | 1 2 3 4 | 1 2 3 4 | 1 2 3 4 5 6 | 1 2 3 4 5 6 | 1 2 3

N. B. — Estimates of temperature made by the present writer.

Legend: Beginning of experiment O; end of experiment V; gradual changes of temperature - - - - - -; sudden changes of temperature ———.
Condition of Carbon: all Cement, Cementite ⁑; partly Cement, partly Hardening ⁎; all Hardening, Martensite, blank.

F — hardened, fine crystalline, porcelanic or quasi-amorphous, characteristic of steel hardened at a proper temperature.

D — hardened, coarse crystalline, characteristic of steel hardened from too high temperature.

After heating to W or any higher temperature, the fracture of the steel will be nearly independent of its thermal treatment prior to this heating: but the fracture of steel which has last been heated to a temperature below W depends greatly upon prior thermal treatment, as the experiments 50 to 55 show.

If the steel in tests 2 and 5 of Experiment 53 and tests 2 and 5 of Experiment 54 had been held long enough at V (690°), the resultant fracture would have been C.

BRINELL'S RULES (modified by the author). — (1) The finer the fracture the better is the condition of the steel; therefore the fractures C, F, H and FH are to be sought, while A, D, H', DH', B and E are to be avoided; of these latter six, B and E are not so bad as the rest.

(2) In forging steel, avoid carrying the temperature needlessly far above W, and continue the forging until the temperature has fallen to a low red (see Experiments 63 and 64, Finishing Temperature).

(3) In hardening steel it is necessary to heat it to W in order that it shall harden: but in order to avoid giving the steel a coarse fracture, and to avoid the injuries which accompany this, the temperature from which it is quenched should be as little as practicable above W.

(4) If, in heating steel in order to harden it, any part of it be heated far above W, the damage done by the overheating is not removed by cooling the steel to W before hardening it, because this cooling does not remove the coarse structure which the overheating has caused. In order to remove these the steel should be cooled below V and then reheated to W. Experiment 55 illustrates this. In the first case the steel was quenched from a temperature far above W. In the second case it was heated to the same temperature and then cooled to W before quenching it, but this did not affect its fracture, which remained coarse crystalline, E. In the third case the steel which had been heated to the same high temperature, was cooled below V, and then again heated to W, and quenched at W. It then had the fine porcelanic

fracture F, and the good quality which accompanies it. Hence the motto, " Do not harden steel on a falling temperature."

CASE-HARDENING. — One instance of this general rule is that when steel has been carburized superficially by heating in contact with carbonaceous matter for case-hardening, it should be cooled completely, or at least below V (Ar_1), then reheated to W, and then quenched. If, after carburizing, the steel is merely allowed to cool to W, and is then quenched, it has the coarse fracture and the consequent poor quality due to the high temperature at which the carburization takes place.

(5) Temperature W may be learned by Metcalf's experiment (Experiment 3, page 12).

(6) Temperature V may be learned by heating one end of a bar of steel above W, and allowing the bar to cool in the dark. As the temperature falls past V (Ar_1) it suddenly rises spontaneously, and the steel is said to recalesce. This recalescence then shows us temperature Ar_1. A more convenient way, however, is that already described in Experiment 2, page 10.

BRINELL'S TEMPERATURE V appears to be Ar_1 (690°), and his W appears to be Ac_{2-3}, and therefore to vary with the percentage of carbon, as shown in Roberts-Austen's diagram, Fig. 27.

REFERENCES: The Author, " The Metallurgy of Steel," pp. 170-176. J. A. Brinell, *Stahl und Eisen,* V, p. 611, 1885: *Jernkontoret's Annaler,* 1885. Coffin, *Trans. Am. Soc. Civ. Eng.,* XV, p. 318. Stead, *Journal Iron and Steel Inst.,* 1898, I, p. 145; 1898, II, p. 137; *Engineering and Mining Journal,* LX, p. 537, Dec. 7, 1895.

Experiment 56

GROWTH OF THE EFFECTS OF OVERSTRAIN. — Overstrain, or straining beyond the elastic limit, itself raises the elastic limit. Moreover, the new elastic limit changes after the overstraining, at first rapidly, then more slowly; and the change is hastened by slight heating.

The following experiment investigates these changes.

Preparation. — 24 pieces of steel wire containing not more than 0.15 per cent of carbon, about 0.033 of an inch in diameter and 3 inches long, are first to have the effects of any initial overstrain removed by annealing. To this end, set them in a piece of

gas pipe about 6 inches long and 1 inch in diameter, closed with a plug at each end, and heat it to dull redness in a muffle furnace. After cooling slowly in the air they are ready for overstraining.

Overstraining. — File each wire flat at one end, so that it can be held firmly in a vise. Grasping the other end with a pair of pincers and holding the axis of the wire straight, twist the wire around its own axis three complete revolutions of 360° each. Before twisting them read the description of the test of lot 2 below.

Testing. — Each wire is to be tested transversely, and the results of these tests are to be recorded, as described in Experiment 8.

The 24 twisted wires are to be divided up into eight lots, which are to be tested as follows:

Lot 1, after twisting, is to be re-annealed by reheating to dull redness (between 600° and 700° C.), and then cooling slowly. This annealing should be done in a closed gas pipe, to prevent oxidation and loss of section.

Lot 2. Each wire of this lot is to be tested as quickly as possible after twisting. To this end twist only one wire of this lot at a time, and test it before twisting the next. Conduct the test as rapidly as is consistent with accuracy.

Lot 3. After twisting, each wire of this lot is to be immersed in boiling water for 10 minutes, cooled, and then tested.

Lots 4 to 8. After twisting, the wires of these lots are to rest undisturbed for the lengths of time indicated below, before testing.

Lot 4	2 hours
" 5	6 "
" 6	1 day
" 7	2 days
" 8	8 "

State concisely your inferences as to the effect of this overstrain, and more particularly as to how this effect is influenced by rest and by slight heating. In this statement cite explicitly the individual results on which you base your several inferences. With this statement present also a tabular statement and a graphical representation of your results.

Experiment 57

REFINING OVERHEATED STEEL, FULL EXAMINATION. (For advanced students.) — 10 like bars of steel containing between 0.35 and 0.45 per cent. of carbon, $^5/_{16}$ inch square and 16 inches long, are to be overheated to 1400° C., and cooled slowly. Determine with a Le Chatelier pyrometer the temperature reached, using the arrangement shown in Figs. 36, 37, and 38. Of these bars, one is to receive no further thermal treatment, and the others are to be reheated severally to a series of temperatures from 20°

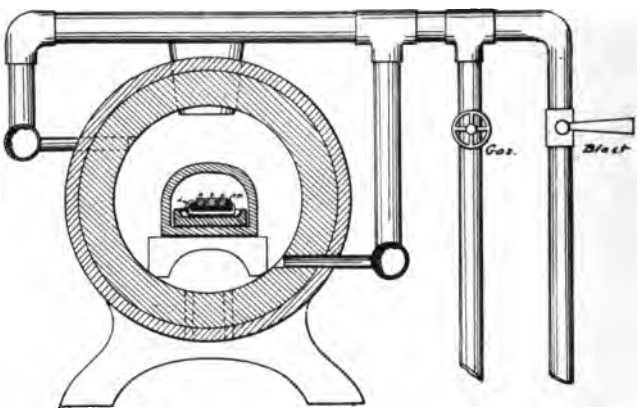


Fig. 36. — Cylindrical Gas Forge, Showing Arrangement of Bars. (Cross-Section.) — Scale 1 = 8.

below $Ac_{2\text{-}3}$ to 20° above $Ac_{2\text{-}3}$, and cooled slowly. To this end these critical points must be determined accurately.

Determine the tensile properties of the series, examine and photograph the microstructure, measure the grain-size, and record it. Examine also the size of the grain as shown in the fracture. Submit a diagram with the temperature reached in reheating as abscissa, and grain-size, tensile strength, elongation and elastic limit as ordinate: also a tabular statement of all your results.

From your results prepare and submit a discussion of the following explanation of the phenomena.

When this steel has been overheated far above the critical range, the network of ferrite which forms during slow cooling

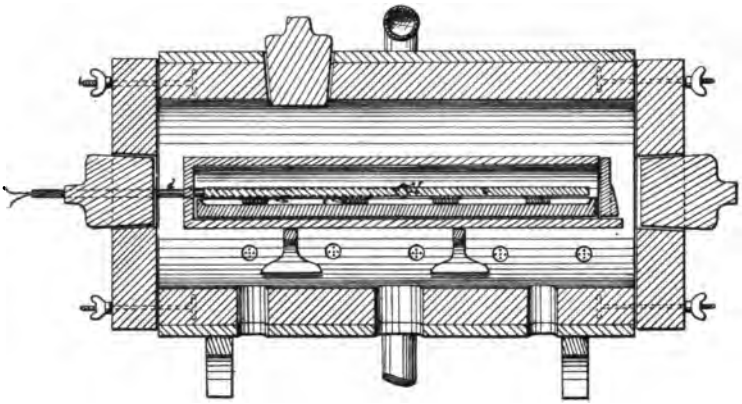


Fig. 37. — Cylindrical Gas Forge, Showing Arrangement of Bars. (Longitudinal Section.) — Scale 1 = 8.

through that range is very coarse. When such steel is again reheated through the critical range Ac_1-Ac_3, the network of ferrite progressively redissolves in the polygons of pearlite which it surrounds, and, by the time Ac_3 is reached, the redissolving of this network has become complete, so that the whole mass is resolved into martensite, a solid solution free from such network. It is this coarse network of ferrite, creating a coarse cleavage system,

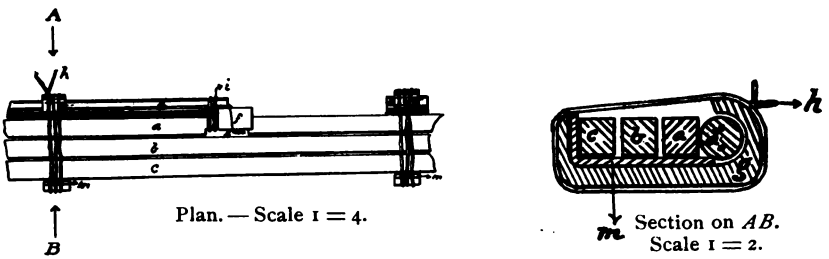


Fig. 38. — Sketch Showing Manner of Binding Bars and Couple in Place.

Legend to Figs. 36, 37 and 38 :

a, notched blank bar.
b, *c*, test bars.
d, double-bored clay tube carrying leads of thermo-junction.
e, thermo-junction.
f, asbestos card.
g, soft steel supports for test bars and leads.
h, string.
i, soft steel wire.
m, asbestos card, separating bars from supports.
o, distance piece.

which leads to the coarse fracture; and it is the removal of this coarse network through the progressive absorption of the ferrite into the surrounding ground mass that removes this coarse cleavage, and the consequent coarseness of structure seen in the fracture. When the steel is again cooled, the martensite is again resolved into polygons of pearlite, surrounded as before by a network of ferrite; but this new network is very fine, if this last heating rose only slightly above the critical range Ac_1-Ac_3, and consequently the cleavage, and the structure of the steel as shown by fracture, are very fine.

Experiment 58

BURNING HYPO-EUTECTIC STEEL, FULL EXAMINATION. — Heat four bars like those of Experiment 57, so hot that they scintillate, allow them to scintillate for 30 seconds, and then cool them slowly. Such steel is said to be "burnt." Two of these bars are to receive no further thermal treatment; the other two are to be reheated to the temperature which you have found in Experiment 57 gives the greatest ductility (or if you have not performed that experiment, then to Ac_3), and again cooled slowly.

Test, examine and report on these bars quite as in Experiment 57.

From your results discuss the following explanation of the phenomena, and cite explicitly any phenomena which support it and any which oppose it.

The scintillation consists in the expulsion of minute particles of metal by a sudden evolution of gas within it. It is probable that this gas is carbon monoxide, generated subcutaneously by the union of the atmospheric oxygen, or of the coating of iron oxide (scale), with the carbon in the metal. When steel is heated so hot as to scintillate, in addition to the coarsening of the grain which any overheating causes, the grains actually yawn apart and crevices form between them. This dehiscence would be a natural result of the sudden evolution of gas which causes the scintillation.

The microstructural changes which accompany the passage upward through the critical range Ac_1-Ac_3 could not be expected to close up actual mechanical separations, actual dehiscences. Hence the failure of these microstructural changes to refine and

restore steel which has actually been thus burnt, although they suffice to refine that which has simply been overheated.

Experiment 59

REFINING OVERHEATED STEEL, BRIEF EXAMINATION. — Repeat Experiment 57, but have your bars 9 inches long, and omit the tensile tests and microscopic examination, and for them substitute the drop-test as described in connection with Experiment 4.

Plot your results with reheating temperature as abscissa, and degree bent on rupture and number of blows endured as ordinates. Submit this, together with a tabular statement of your results, and a brief enunciation of the law to which they point. In particular, state to which, if any, of the critical points Ac_1, etc., the refining temperature W corresponds.

Experiment 60

BURNING HYPO-EUTECTIC STEEL, BRIEF EXAMINATION. — degree bent on rupture and number of blows endured as ordinate. the tensile tests and microscopic examination, and for them substitute the drop-test as described in connection with Experiment 4.

State concisely your inferences from this experiment when compared with Experiment 59.

Experiment 61

INFLUENCE OF $T^{MAX.}$ ON THE THOROUGHNESS OF REFINING, FULL EXAMINATION. — 12 bars of steel of about 0.80 per cent of carbon, 16 inches long and five sixteenths inch square are to be prepared. Of these two are to be heated to each of the following temperatures and cooled slowly under like conditions.

1000° C.
1250° C.
1300° C.
1350° C.
1400° C.

One bar of each of these pairs is to receive no further thermal treatment; the other is to be reheated to slightly above $Ac_{1\text{-}2\text{-}3}$ (say to 740°), and cooled slowly.

Determine the tensile properties of the series, examine and

photograph the microstructure, measure the grain-size, and record it. Examine also the size of the grain as shown in the fracture. Submit a diagram with the temperature reached in reheating as abscissa, and grain-size, tensile strength, elongation and elastic limit as ordinate: also a tabular statement of all your results.

Enunciate briefly the law governing these phenomena, and offer an explanation of them.

Experiment 62

INFLUENCE OF $T^{MAX.}$ ON THE THOROUGHNESS OF REFINING, BRIEF EXAMINATION. — Repeat Experiment 61, but with bars 9 inches long. Omit the tensile tests and microscopic examination, and for them substitute the drop-test as described in connection with Experiment 4.

State briefly the law to which your results point.

Experiments 63 and 64

FINISHING TEMPERATURE, GENERAL DIRECTIONS. (For advanced students only.) — The ductility of steel in general is lessened by undisturbed slow cooling from high temperatures. If, after the steel is cooled from a high temperature to a lower one, it is deformed, as for instance by rolling, the injury is lessened, and the structure and properties of the steel are more nearly those which it would have had, had it been heated originally only to this lower temperature. In short, within limits, the lower the temperature at which the rolling of the steel ends, i. e., the lower the finishing temperature, the more ductile is the steel.

The object of the investigation is to determine quantitatively the effect of different finishing temperatures upon the tensile strength, elastic limit, ductility and microstructure of the steel selected. To that end bars of the steel to be studied should be heated to a given high temperature, sufficient to cause marked injury, and should then be cooled undisturbed. After such slow cooling has reached a certain temperature, different for each of the several bars, the steel should undergo a definite reduction by rolling, — the same for all the bars, — and should then cool slowly and completely without further disturbance. The difference between the several bars will then be due to the differences in finishing temper-

ature. The bars are then to be tested tensilely, and their microstructure, especially the average grain-size, is to be determined, so that we may learn what microstructural indications correspond to these different finishing temperatures. Then plot curves with finishing temperature as ordinate and tensile strength, elastic limit, elongation, contraction of area, and average grain-size as abscissa.

Before the experiment proper determine the heating and cooling curves of the steel very accurately.

Of the finishing temperatures, i. e., those to which the bar is to be cooled before rolling, a large proportion should be taken

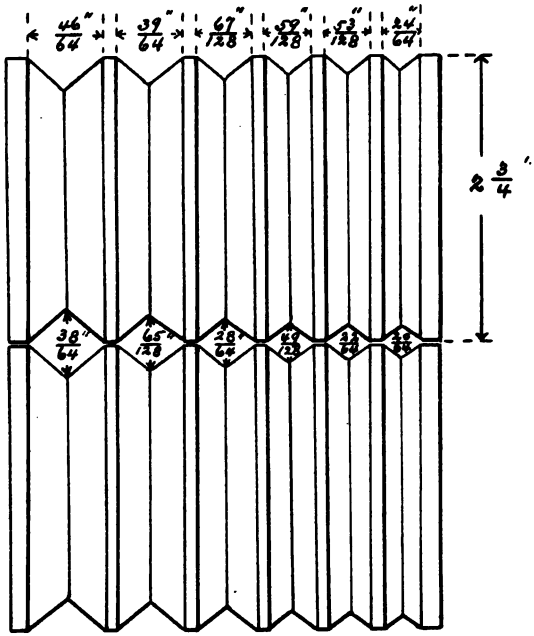


Fig. 39. — Rolls for Finishing Temperature Experiments (Nos. 63 and 64).

in the immediate vicinity of the critical points, because we may expect that the effect of changes in the finishing temperature will itself change on passing them. In determining them, the cooling should be extremely slow, for the reason that in your rolling experiments you will use a very slow cooling, and that the position of the critical points, particularly that of Ar_1, is affected by the rate of cooling, as is to be expected. The slower the cooling

the higher is Ar_1, i. e., the nearer does it come to the theoretical critical temperature.

Each bar before heating should be stamped with its series number and its own individual number, and these same numbers should appear in the tabular statement of your results. The fragments of your bars should be preserved in a box of suitable size labeled with your name and the date.

Size of Bars and Reduction. — These must depend upon the rolls and power at hand, and will have to be determined for each individual case. So far as possible they should be the same throughout a given experiment. A convenient size is $^7/_{16}$ inch square, and rolls suitable for rolling bars of this size are shown in Fig. 39.

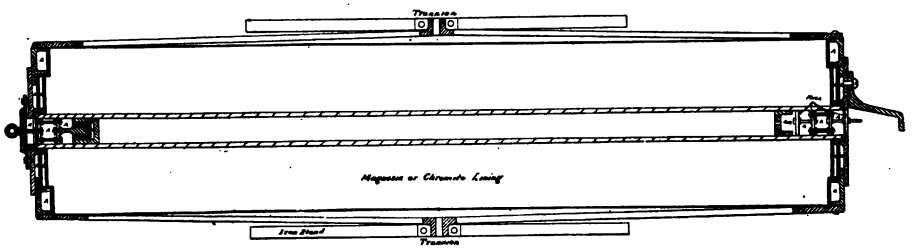


Fig. 40. — Electric-Resistance Tubular Muffle Furnace (designed by the author with the assistance of Mr. W. S. Hadaway, Jr.). The furnace is heated by the resistance of four platinum wires wrapped spirally around grooves in the outside of the tube in multiple. It rotates about its own axis and also about the trunnions shown. — Scale 1 = 12.

Mode of Heating. — In each heating and cooling two or four like bars should be placed side by side, with a notch filed in one, as shown in Fig. 38. Each bar under observation should be allowed to cool in this position until it reaches the temperature at which it is to be rolled, so that its finishing temperature may be accurately known. It is then to be drawn and to be passed once through the rolls.

The temperature during heating and cooling has to be accurately recorded as often as necessary, and in no case at intervals of more than 15 minutes. On approaching the maximum temperature frequent observations should be taken, so that you may be confident that you have not overstepped the maximum aimed at.

Graduate students may use the electric-resistance furnace for their heatings; others will use the gas forge.

DIRECTIONS AS TO THE ELECTRIC FURNACE (Figs. 40 and 41). — In using this you must be very careful never to allow the steel or the scale from the steel to fall upon the clay tube, because it is very quickly destroyed by iron scale. A porcelain tube may be put inside of the clay tube to protect it, or it may be protected at relatively low temperatures by sheet asbestos. But if asbestos is used, it must be scraped out every two days and replaced with fresh, lest the iron scale corrode the clay tube.

FINISHING TEMPERATURES TO BE SELECTED. — These should comprise, (1) every 50° C. between the temperature to which

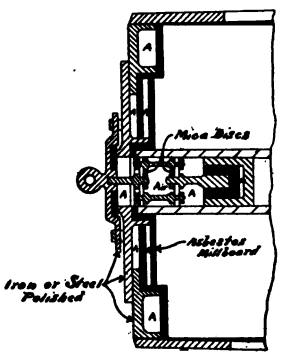


Fig. 41. — End of Electric-Resistance Furnace, Enlarged. — Scale 1 = 6.

the steel is heated (T^{max}) and 700° C., or as much lower as it is found practicable to give the reduction desired, and (2) every 15° for a range of 75° on either side of the several critical temperatures. In using higher carbon steels it may be well to make the reduction by rolling at the lower finishing temperatures slighter than at the higher ones, because the steel is so much harder when cooler.

Experiment 63

FINISHING TEMPERATURE FOR RAIL STEEL. — Follow the foregoing instructions, using steel of between 0.35 and 0.45 per cent of carbon (rail steel) and 1400° C. as T^{max}.

Experiment 64

FINISHING TEMPERATURE FOR STRUCTURAL AND BOILER PLATE STEEL. — Follow the foregoing instructions, using steel of between 0.18 and 0.24 per cent of carbon (structural and boiler plate steel) and 1450° C. as T^{max}.

REFERENCES: "Metallurgy of Steel," Howe, Sections 295 to 298 inclusive. *Journal Iron and Steel Institute,* 1897, Vol. I, p. 527; 1898, Vol. I, p. 145, Vol. II, p. 137. *Stahl und Eisen,* Vol. XVII, p. 51. *The Iron Age,* Vol. LXVI, Dec. 20, 1900, p. 16. Sauveur, *Trans. Am. Inst. Mining Engineers,* Vol. XXII, p. 546. *Engineering and Mining Journal,* LX, p. 537.

Experiments 65 to 69 — Microstructure

Experiment 65

MICROSTRUCTURE OF PARTLY CONVERTED BLISTER STEEL. — This experiment is only for those who have already acquired considerable expertness in the use of the microscope.

The material to be examined is "partly converted blister steel," i. e., the product resulting from heating wrought iron or low carbon steel in contact with charcoal, as in the cementation process, but only long enough to carburize it part way through, leaving a central core of approximately the original composition. Such steel may be had by withdrawing a bar from a cementation furnace after it has been heated for about one-third the normal time.

Determine the distribution of carbon in the steel by direct analysis, in successive layers, each one $^1/_{16}$ of an inch thick, from the outside to the center. Now polish a piece of the steel so taken as to show a section of the bar from edge to edge. Develop the pearlite in this, estimate the percentage of pearlite and of ferrite or cementite at different depths, and compare these results with the percentage of carbon found at the same depths. Where there is 0.80 per cent of carbon, the steel should consist of pearlite alone; where there is more carbon than 0.80 per cent (hyper-eutectic steel) the pearlite should be accompanied by cementite. Where there is less carbon (hypo-eutectic steel) the pearlite should be accompanied by ferrite.

REFERENCE: Osmond: "'Méthode Générale pour l'Analyse Micrographique des Aciers au Carbone" (Société d'Encouragement, Extract from *Bulletin,* May 1895). See preferably the reprint in the "Contribution à l'Étude des Alliages" (Société d'Encouragement, 1901), page 277.

Experiment 66

DISTRIBUTION OF MARTENSITE. — A piece of the steel used in Experiment 65, cut so as to show a section from edge to edge, and preferably not more than ⅛ of an inch thick, so as to cool very rapidly, is to be heated to 1300° and quenched as rapidly as possible in a freezing mixture, such as iced brine or better an intimately and quickly made mixture of 70 parts of snow or very finely divided ice and 100 parts of powdered, previously cooled, calcium chloride. The freezing mixture should be at least as cold as —10° C., and at least one litre of it should be used. Quenching in liquid air direct does not cool the steel very rapidly, because the steel immediately coats itself with a layer of gaseous air, a very poor conductor of heat.

Examine with the microscope, and describe and photograph the appearance of the martensite at different depths.

To lessen oxidation the steel may be heated in the closed electric-resistance crucible furnace, in which a lump of charcoal may first be placed. A stout wire should be attached to the steel, to facilitate grasping it quickly when the furnace is opened for its removal.

Experiment 67

DISTRIBUTION OF MARTENSITE.— Prepare and quench another slice of the same steel exactly as described in Experiment 66, except that the steel is first to be heated to 1000° C., cooled slowly (e. g., in the furnace itself) to 750°, and then quenched from this temperature. Examine the structure under the microscope, and especially note the changes of structure that occur in the neighborhood of carbon of 0.45 per cent and of carbon of 0.85 per cent.

Compare Roberts-Austen's diagram (Fig. 27). Also Sauveur (*Trans. Am. Institute Mining Engineers,* XXVI, p. 883).

Note that the layers containing less than 0.43 per cent of carbon have been quenched from between Ar_1 and $Ar_{2\text{-}3}$; that those containing from 0.43 to 0.85 per cent have been quenched from above Ar_3, and those containing more than 0.85 per cent of carbon have been quenched from between $Ar_{2\text{-}1}$ and Ar_3.

Experiment 68

METCALF'S EXPERIMENT ELABORATED. — Proceed as in Experiment 3, but use a thin bar of steel of 0.70 per cent of carbon, preferably about ½ x ⅛ of an inch, so that the cooling may be more rapid, and do not nick it, lest the fractured surfaces be too small for convenient examination. Further, let the quenching be in iced brine or other freezing mixture.

After quenching the bar, breaking it up into short fragments, and noting the changes in their fracture as in Experiment 3, polish and etch various points along its length, especially in the region where hardening begins. Grind away as little as possible of the outer surface in preparing the pieces for polishing, because it is in the very skin only that the cooling is extremely rapid.

Compare the transition from the unrefined to the refined fracture with the transition from pearlite to martensite in the polished sections, and report in writing your observations. Also report whether you detect under the microscope any increase of grain-size in the parts which had been hottest, corresponding to the increase in grain-size in the fracture.

Experiment 69

METCALF'S EXPERIMENT ELABORATED. — Repeat Experiment 68 with the exception that the carbon is to be between 0.50 and 0.60 per cent, and that the steel is to be cooled slowly instead of being quenched.

Compare the fractures with Brinell's chart.

Now polish and examine under the microscope two points upon each of the fragments. You will find that the steel consists of pearlite with a network of ferrite, and that the size of the network increases with the temperature which had been reached in heating, i. e., with T^{max}, in that part which has been heated above the critical range.

Report your results in writing with your own brief statement of the quantitative relation between the grain-size and T^{max}.

GROUP 6. — NON-FERROUS METALS

COPPER

Experiment 70

COPPER SELECTING PROCESS. — (1) *Preparation of Impure Matte.* — Melt in a graphite crucible 500 grammes of copper matte containing between 65 and 78 per cent of copper, and when it is thoroughly molten drop in successively 1 per cent each of arsenic, antimony and bismuth, and 0.1 per cent of silver. Pour the matte into an iron mould and determine the copper, arsenic, antimony, silver and bismuth.

(2) *The Selecting Process.* — Set 100 grammes of the pulverized matte in a graphite crucible, and upon it put 50 grammes of shot copper of known purity; cover the copper with half an inch of powdered charcoal; lute the crucible, and heat it to at least 1200°. Pour the molten mass into a deep iron mould; when cold separate the resultant buttons of copper and matte; and weigh and determine in each the copper and each of the four foreign elements which have been added.

The matte and the metallic copper will remain as two distinct bodies, for the reason that in the dry way no copper sulphide forms containing more copper than cuprous sulphide (Cu_2S, copper 79.86 per cent, sulphur 20.14 per cent).

The sulphur present unites with copper up to this ratio, and any excess of copper remains in the metallic state and does not coalesce with the overlying cuprous sulphide or matte. Metallic copper thus separated is called "copper bottoms," and the ingot copper made by refining it is called "best selected copper."

This experiment will show to what degree these elements separate themselves from the copper sulphide and unite with the metallic copper, thanks to their lower affinity for sulphur. This separation of these impurities from the copper matte, and their concentration in the "copper bottoms," even if the quantity of these bottoms is very small, are the basis of the Welsh "selecting" process, and play an important part in the other dry processes for sulphide copper ores.

Report your results in writing with your own brief quantitative statement of (1) the degree to which the several elements

separate and pass into the bottoms, and (2) of the relative completeness of separation of the several elements.

Compare Third Report of the Alloys Research Committee, Appendix 1, and Keller (*Trans. Am. Institute Mining Engineers,* XXVIII, pp. 127-159 and 816-840).

Experiment 71

SPELTER TEST. — The fracture of Muntz metal (brass with copper 60 per cent, zinc 40 per cent) gives an indication of the purity of the copper from which it is made. If the copper is very pure, the fracture of the Muntz metal is fine and hackly, but even small quantities of impurities cause a columnar fracture.

Prepare three lots of copper containing respectively 5 per cent of arsenic, 5 per cent of antimony and 5 per cent of bismuth. The quantity of each to be prepared is that required for making the exhibits enumerated below, plus an excess of 25 per cent.

Preparation of Copper. — Melt pure shot copper in a graphite crucible under powdered charcoal, and when thoroughly melted drop in five per cent of metallic arsenic, and stir with a stick of wood (not iron). Granulate the copper by pouring the metal into water.

Prepare the antimony and bismuth copper in the same way.

Preparation of Muntz Metal. — Melt in a graphite crucible under powdered charcoal enough copper to yield the products enumerated below, plus 25 per cent excess. When well melted, stir into it enough lump zinc to yield Muntz metal of 60 per cent copper and 40 per cent zinc, assuming that 42 per cent zinc must be added to 58 per cent copper on account of volatilization of the zinc, in order to yield the Muntz metal ratio of 40 : 60.

When thoroughly melted and superheated for thirty minutes, and while still covered with powdered charcoal, drop in enough of the arsenical copper to give 0.10 per cent arsenic in the whole mixture, and pour one ingot. Then without replacing in the furnace add enough more arsenical copper to raise the arsenic in the remaining calculated weight of Muntz metal to 0.25 per cent, and pour a second ingot. The crucible may now have to be reheated to prevent the copper from freezing. To the metal remaining in the crucible add enough arsenical copper to give the remaining brass 0.50 per cent of arsenic, and pour a third ingot,

and in the same way from the still remaining brass (if possible, without replacing in the furnace) prepare ingots with one per cent and with two per cent of arsenic.

Operating in the same way, start with a fresh lot of Muntz metal, and cast first an ingot of pure brass and then successive ingots with bismuth, .02, .05, .10, .20 and .40 per cent.

Starting in the same way with the third lot of Muntz metal, prepare ingots with antimony, .01, .02, .05, .10 and .20 per cent.

These ingots are to be nicked, broken, assembled and photographed. Assemble the photographs on cardboard, mark the composition of each, and hand them to the officer in charge, together with your own brief statement (1) of the influence of each of these impurities on the fracture, and (2) of the relative influence of the several impurities on the fracture.

All products throughout this investigation are to be weighed, and the percentage of arsenic in the arsenical copper, of antimony in the antimonial copper, and of bismuth in the bismuth copper, are to be determined. If possible the copper and impurities in each of the resultant brasses also are to be determined.

REFERENCE: Sperry (*Trans. Am. Institute Mining Engineers.* XXVIII, pp. 176 and 427).

Experiment 72

COPPER UNITES WITH SULPHUR IN PREFERENCE TO OXYGEN. — To a ferrous sub-silicate slag, such as a puddling slag of known composition, add enough cuprous oxide and silica to bring the composition of the whole to equal equivalents of cuprous and ferrous singulo-silicates, $2Cu_2O,SiO_2$ and $2FeO,SiO_2$. Prepare enough to represent 100 grammes of metallic copper. Grind the whole to pass through a screen of 16 meshes to the linear inch, and place it in the bottom of a graphite crucible about 2½ inches in diameter. Upon the silicate place ferrous sulphide in such amount that its sulphur, if all utilized, would theoretically suffice to form cuprous sulphide with 125 grammes of metallic copper; i. e., provide thus 25 per cent excess of sulphur over that theoretically required to convert the whole of the copper present into cuprous sulphide. Lute the crucible tightly and heat to about 1500° C. for thirty minutes. Withdraw the crucible and pour the contents into an iron mould. Separate the resulting slag and matte from each other, and determine the percentage of iron and copper

in each. You will find most of the copper in the matte, and most of the iron in the slag.

This shows that when iron and copper are melted in the presence of sulphur and oxygen, the copper unites by preference with the sulphur, and the iron with the oxygen.

If, through any deoxidizing action, more copper be deoxidized than can form cuprous sulphide (Cu_2S) with the sulphur available, then this excess of unoxidized copper above the Cu_2S ratio separates out in the metallic state, because no copper sulphide forms in the igneous way containing more copper than cuprous sulphide, and such metallic copper does not coalesce with the matte. In this way it may happen that a button of metallic copper will be found, in addition to the slag and matte. (See Experiment 70.)

Experiments 73 to 78

REFERENCES: H. M. Howe, "The Metallurgy of Argentiferous Copper Compounds," *Report of the Director of the Mint upon the Production of the Precious Metals in the United States during the calendar year* 1883 (Burchard), p. 774; "Elements of Metallurgy," John Arthur Phillips; "Sulphuric Acid and Alkali," Lunge, I, p. 625; *Chem. News,* XXIII, p. 26, and XXXI, p. 165; "Wagner's Jahresbericht," 1872, p. 152; Dingler, CCIV (p. 288), CCXIV (p. 466), CCXV (pp. 54 and 229), CCXIX (p. 323), CCXXXI (p. 323); *Zeit. B. H. & Sal.,* 1877, p. 156.

CHLORIDIZING ROASTING, GENERAL DIRECTIONS. — The copper of some copper ores can be converted into cupric chloride, $CuCl_2$, by roasting the ore with common salt, provided that there is some means of liberating the chlorine of that salt. The sulphuric anhydride, SO_3, formed by the oxidation of a small quantity of sulphur present in the ore, is an effective agent for liberating this chlorine, and through it the copper may be chloridized by the reaction

$$2\,NaCl + SO_3 + CuO = CuCl_2 + Na_2SO_4.$$

In practice this principle is used in the Longmaid (Henderson) process. In this process, after pyrites containing a small quantity of chalcopyrite (technically called "cupreous pyrites"), and very nearly free from gangue, has had nearly all its sulphur extracted, for conversion into sulphuric acid, by an oxidizing roasting (in pyrites kilns in lump form or otherwise), it is next coarsely ground, mixed with some 7 per cent of common

salt, and re-roasted, this time at a low temperature in long reverberatory roasting furnaces. The sulphuric anhydride formed by the small quantity of sulphur left after the first or kiln roasting suffices to decompose the salt, and enables its chlorine to unite with the copper present.

The copper chloride thus formed can then be dissolved out with water, and after removal to an appropriate precipitating tank, its copper can be precipitated with scrap iron or milk of lime, and thus recovered.

For the commercial success of the process it is important (1) to use the minimum quantity of so expensive a reagent as common salt, and (2) to extract as much as permissible of the sulphur of the pyrites in the first or kiln-roasting of the ore, in order that the yield of sulphuric acid should be as great as possible; so that in the kiln-roasted ore which is to undergo the chloridizing roasting there should be no more sulphur left than is really necessary for the purposes of that roast, i. e., for the liberation of the chlorine needed.

Thus it is important to know the minimum quantity of salt and the minimum quantity of sulphur needed to permit thorough chloridizing of the copper. The following experiments address themselves to these points, and enquire as to the best temperature.

General Plan. — The material to be chloridized is cupreous pyrites which has first undergone a very thorough oxidizing roast as described in Experiment 6, in a 20-pound lot. The oxidizing roast should be so prolonged that not more than 3 per cent of sulphur is left in the ore. Then the several chloridizing roasts are made with different percentages of sulphur (in the form of iron pyrites), and of salt, in order to learn the influence of different proportions of these reagents on the thoroughness of the chloridizing. In this way the material treated in all the chloridizing roasts will be alike, except in the proportion of salt and iron pyrites thus introduced.

The cupreous pyrites treated should contain between 2.5 and 5 per cent of copper, and not over 15 per cent of gangue. Should such an ore not be at hand, an artificial ore may be made by mixing chalcopyrite and pyrites in proper proportions; they should be very finely ground and intimately mixed.

Three sets of chloridizing roasts are to be made. The first set aims to determine the best roasting temperature; the second

the influence of different proportions of raw pyrites added; and the third the minimum quantity of salt permissible. Then from the data thus obtained the most economical procedure is to be calculated. Then two further experiments examine the influence of sulphur and ferric oxide on the chloridizing.

The chloridizing roasts are to be made in the oven-roasting furnace (Fig. 9), and each treats a one-pound lot of pre-roasted ore, ground to pass a screen of 16 meshes to the linear inch. In each roast take samples every fifteen minutes for the first hour, and every thirty minutes for the rest of the afternoon, carrying on the roasting from 2:30 to 5 P.M. in each case. In each sample determine (1) the total copper, (2) the copper soluble in boiling water, and (3) copper soluble in hydrochloric acid, as in Experiment 6. Throughout every roast record the temperature and stir the ore thoroughly every fifteen minutes, stirring it so thoroughly as to shift the ore which is at the further end of the furnace toward the nearer end, and every hour make a complete shifting from one end of the furnace to the other.

Three diagrams are to be prepared, one for each of these three sets of roasts. In each plot time as abscissa and the percentage of the copper present which is soluble in boiling water as ordinate; and show the progress of each roast by drawing a curve through the points which represent it. Plot your results immediately after each roast for your own guidance in making the following roast.

Experiment 73

FOUR ROASTS TO FIND THE BEST TEMPERATURE (ROASTS 1 TO 4). — In each of these four roasts use 15 per cent of salt, and add enough raw pyrites free from copper to form, together with the sulphur initially present, 2.5 parts of sulphur to each part of copper present by weight. Roast the first lot at 435° C., keeping the temperature as nearly constant as practicable. Roast the second lot at 500° in the same way, and from the results obtained from these two temperatures select a third temperature which you think most likely to give the best results. From the results obtained in these three roastings select a fourth temperature at which you now think you will get the best results.

In every case "best results" means the largest amount of copper made soluble in boiling water in the shortest time.

Experiment 74

To Find the Best Percentage of Raw Pyrites to Add (Roasts 5 to 8). — Make four different roasts, all of them at the temperature which you have now found gives the best results. Roast your first lot with enough raw pyrites to form, with the sulphur present in the cupreous pyrites, 1.5 parts for each part of copper present, by weight. Roast your second lot with 2.5 parts of sulphur per part of copper, and your third with 0.75 parts of sulphur per part of copper. From the data thus reached make your own inferences as to the proportion of pyrites which will give the best results, and make your fourth roast with this proportion.

Experiment 75

To Find the Minimum Quantity of Salt (Roasts 9 to 12). — Make four roasts, in each of them using the temperature and the proportion of raw pyrites which you have now found to give the best results, varying the proportion of salt used. Make your first roast with 15 per cent of salt, your second with 10 per cent; your third with the proportion of salt which, judging from the results of the first two roasts, should be about the minimum which will thoroughly chloridize the copper; and then make your fourth roast with a proportion of salt similarly based on the results of the first three roasts.

Experiment 76

Calculation to Find the Most Economical Conditions. — Assume the following costs and values:

Iron pyrites (cost)	$4.00 per ton
Cupreous pyrites (cost)	8.00 " "
Salt (cost)	3.00 " "
Roasting-cost per hour (including fuel, labor, repairs and proportion of general expenses) per ton of "material treated" (including both iron and cupreous pyrites and salt in the weight of material treated)	0.15 per ton
Copper made soluble by chloridizing (value)	0.08 per lb.

From these and the results which you have obtained calculate what proportion of salt and of pyrites should be used, and at what point the roast should be ended, to yield the greatest profit,

(1) per ton of cupreous pyrites roasted, and

(2) per square foot of hearth of roasting furnace per 24 hours, assuming that there are 15 pounds of ore on each square foot of hearth at any given instant.

Experiment 77

To Show the Influence of Sulphur in Chloridizing. — Prepare synthetically a one-pound lot of artificial ore, of the same composition as the material treated in roast 12 (Experiment 75), except that it is free from sulphur. This may be done by mixing in proper proportions (1) cupric oxide, (2) ferric oxide, (3) salt, and (4) silica to represent the gangue. Roast as in roast 12, and determine the condition of the copper as before. From the results enunciate your inference as to the importance of having sulphur present.

Experiment 78

To Show the Influence of Ferric Oxide in Chloridizing. — Proceed as in the last experiment, except that the artificial ore is to be as nearly free from iron oxide as is consistent with having a like copper-bearing mineral, chalcopyrite, under treatment. The artificial ore may be made by mixing in proper proportions, (1) pure chalcopyrite raw, (2) pure chalcopyrite roasted, (3) salt, and (4) silica in quantity sufficient to represent both the initial gangue and the ferric oxide of roast 12, per cent for per cent, after allowing for the ferric oxide introduced in the present roast by the chalcopyrite.

Roast as in roast 12, and determine the condition of the copper as before. From the results enunciate your inferences as to the part (if any) played by ferric oxide, and your explanation.

Experiments 79 to 85

Illustrating the Ziervogel Process.

REFERENCES: R. H. Bradford, "The Reactions of the Ziervogel Process and their Temperature Limits," *Trans. Am. Inst. Min. Engineers,* Washington Meeting, 1902, to appear. — Steinbeck, "Chemisch-Analytische Untersuchungen über die Ziervogelsche Extractionsmethode," Halle, 1862. — The Author, "The Metallurgy of Argentiferous Copper Compounds," *Report of the Director of the Mint upon the Production of the Precious Metals in the U. S.,* 1883, p. 753. — Kerl, "Grundriss der Metall-

hüttenkunde," 1881, p. 352. — Rivot, "Principes Généraux du Traitement des Minérais Métalliques," 1871, I, pp. 370-425. — Egleston, "Mineral Resources West of the Rocky Mountains," 1876, pp. 379-386, and *Trans. Am. Inst. Min. Engineers,* IV, p. 285.

Principle of the Process. — Silver is recovered from argentiferous copper matte by so roasting it as to convert the silver

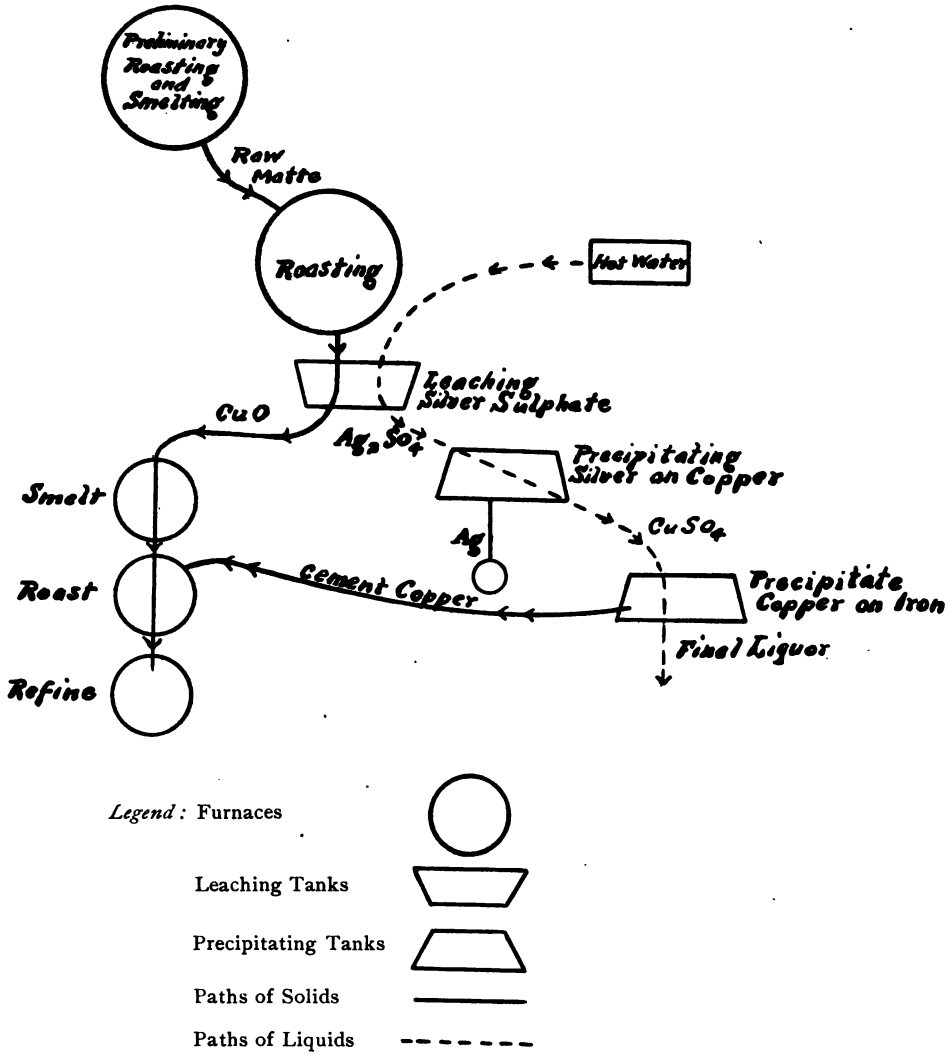


Fig. 42. — Tree of the Ziervogel Process.

into silver sulphate, and then dissolving this sulphate out by means of hot water. From the silver sulphate solution the silver is recovered by precipitating it as metallic silver by means of metallic copper,

(1) $Ag_2SO_4 + Cu = CuSO_4 + 2\,Ag$

When silver sulphide is roasted by itself, only a small fraction of its silver passes into the soluble condition of sulphate, much of it passing into the metallic state, in which it is insoluble in the acidulated hot water used as a solvent in the Ziervogel process. If, however, it be exposed during roasting to nascent sulphuric anhydride (SO_3), a very much larger fraction of the silver passes into the state of sulphate, and thus becomes soluble, i. e., recoverable by this process. This sulphuric anhydride is in practice obtained by the dissociation by heat of cupric sulphate (reactions 3 and 4), which in turn is formed by the oxidation of the cuprous sulphide of the matte itself (reaction 2).

(2) $Cu_2S + 5\,O = CuSO_4 + CuO$

(3) $2\,CuSO_4 = CuO,CuSO_4 + SO_3$ (at 655° C.)

(4) $CuO,CuSO_4 = 2\,CuO + SO_3$ (at about 700° C.)

This formation of cupric sulphate is itself stimulated by the evolution of sulphuric anhydride from the dissociation by heat of the iron sulphates, which in turn are formed by the oxidation of the iron sulphide of the matte. Hence the mattes to which the process is applied are usually ferruginous.

Iron and normal cupric sulphates are dissociated by heat at about 590° and about 655° C. respectively. In thus dissociating normal cupric sulphate changes to basic cupric sulphate and sulphuric anhydride by reaction (3), and at about 700° C. the resultant basic sulphate dissociates by reaction (4), yielding cupric oxide and sulphuric anhydride.

The silver sulphate formed by reaction (1) itself dissociates, yielding metallic silver, at about 870°. Therefore to convert the silver into sulphate the roast must be at a temperature above 655° and preferably above 700° in order to get the needed sulphuric anhydride; yet the temperature should not reach 870°, lest the silver sulphate formed in turn should dissociate, yielding metallic silver, which is insoluble in the solvent used, hot water. In brief, the temperature limits are very narrow.

The finely divided silver resulting from the dissociation of the silver sulphate may itself be re-sulphatized by nascent sul-

phuric anhydride (reaction 5); and no doubt this re-sulphatizing does occur in commercial roasts. (See Experiment 84.)

(5) $CuSO_4 + 2\,Ag + O = CuO + Ag_2SO_4$

The roast must be prolonged not only until the silver has been sulphatized, but also until any copper present as cuprous oxide has been brought to the state of cupric oxide; for the reason that if any cuprous oxide remained in the roasted matte when we later attempt to dissolve its silver sulphate by hot water, this cuprous oxide would re-precipitate within the matte the silver thus dissolved, and thus our attempt to dissolve out the silver would be defeated.

(6) $Cu_2O + Ag_2SO_4 = CuSO_4 + CuO + 2\,Ag$

The sulphides which are to be roasted in Experiments 79 to 82 must in every case be ground so as to pass through a screen of 60 meshes to the linear inch, and the temperature should be controlled by means of the Le Chatelier pyrometer, the couple of which should be imbedded in the roasting matte, as in Experiment 6. The upper surface of the matte may become considerably hotter than the layers below, especially during any rapid rise of temperature or rapid oxidation of sulphur. Therefore, the thermo-junction should be kept very near the upper surface of the matte and should be so placed as to be readily visible and adjustable.

Experiment 79

Ferrous Sulphate for Sulphatizing Copper. — In each of two adjoining pans roast 200 grammes of cuprous sulphide, adding to that in one pan 200 grammes of hydrated ferrous sulphate (copperas), and to that in the other pan 200 grammes of silica sand. There should be free access of air and frequent stirring, and the temperature should be raised very gradually, especially from 475° up to 850°. At about 500° the cuprous sulphide becomes pasty, or it may even melt if it is heated by itself; but if, as just directed, it is mixed with an equal weight of silica sand this difficulty is lessened. Yet in any event this tendency should be held in check by making the rise of temperature from 475° to 550° very slow, and by stirring very actively while passing through this range. The passage from 550° to 850° should occupy at least two hours. At every fifty degrees from 300° to 850°, and also at 625°, 675° and 725°, take a sample from each pan and

determine the percentage of the total copper present as sulphide, sulphate and as oxide respectively, as described in Experiment 6. The percentage of the total iron present as sulphate also should be determined by leaching with water.

This experiment will show first how the sulphatizing of the copper is influenced by the evolution of sulphuric anhydride from the dissociation of iron sulphate, and second how the resultant copper sulphate is decomposed into basic sulphate by superheating above 655°, and this in turn into cupric oxide by superheating above 700°.

Experiment 80

SULPHATIZING ACTION OF FERROUS SULPHIDE. — Repeat Experiment 79, substituting ferrous sulphide for ferrous sulphate, and adding 200 grammes of silica to each pan to prevent too rapid rise of temperature when the sulphides ignite. This will show how ferrous sulphide in roasting yields iron sulphate, which in turn stimulates the sulphatizing of the copper.

Experiment 81

SILVER SULPHIDE WHEN ROASTED YIELDS METALLIC SILVER AND SILVER SULPHATE. — Heat two grammes of finely pulverized silver sulphide in a porcelain crucible in the electric-resistance crucible furnace, to 800°, with moderate access of air. Expose it to temperatures between 700° and 825° for one hour. Determine the silver sulphide present, if any, in the residue, by dissolving it out with acidulated hot water. Examine the residue under the microscope, and describe it. If you find that it consists of metallic silver, then calculate what percentage of the total silver has passed to the state of sulphate, and what to the metallic state.

Experiment 82

THE DISSOCIATION OF CUPRIC SULPHATE STIMULATES THE SULPHATIZING OF SILVER. — Repeat Experiment 81, mixing in with the silver sulphide 5.5 grammes of hydrated cupric sulphate, and as before calculate the percentage of silver which has passed to the metallic and the sulphate states respectively. State briefly your inferences as to the part played by cupric sulphate here.

Experiment 83

THE SPANGLE REACTION. — The object of this experiment is to show how the presence either of cuprous oxide or of magnetic oxide of iron precipitates the silver which has been dissolved as sulphate.

Take four lots of silver sulphate of 0.10 gramme each. Mix the first with one gramme of finely pulverized cuprous oxide, the second with one gramme of finely pulverized cupric oxide, known to be free from cuprous oxide, (e. g., that made by decomposing cupric sulphate), the third with one gramme of finely pulverized magnetic oxide of iron, and the fourth with one gramme of anhydrous ferric oxide or an equivalent quantity of hydrated ferric oxide. Put each in a test-tube with 20 c.c. of boiling water; shake each up and examine the suspended mass.

The sample which is accompanied by cuprous oxide will show bright spangles of metallic silver, showing that this oxide re-precipitates the silver which had dissolved as sulphate. The sample accompanied by magnetic oxide may show a few such spangles, showing that magnetic oxide re-precipitates silver in this way, but much less energetically than cuprous oxide.

The lot accompanied by cupric oxide should show no such spangles, showing that the whole of the silver remains dissolved as sulphate.

Filter off the liquor and determine the silver in the filtrate to check these phenomena.

Oxides of iron intermediate between true magnetic and ferric oxides, i. e., between Fe_3O_4 and Fe_2O_3, may be strongly attracted by the magnet and yet may not have any important influence in precipitating the silver. Therefore, the mere fact that the roasted ore can be attracted by the magnet is no proof that true magnetic oxide is present, nor that the silver will re-precipitate in the matte on attempting to dissolve it.

Experiment 84

SULPHATIZING METALLIC SILVER BY FERROUS SULPHATE. — Heat 0.10 gramme of silver sulphate mixed with 2 grammes of silica sand at 925° for thirty minutes. Cool and determine the silver sulphate remaining, by leaching with hot water and titrating. Heat a second lot in the same way, then cool it to about

700° and mix in one gramme of ferrous sulphate, keeping the temperature for fifteen minutes as near as practicable to 700°. After cooling determine the silver present as sulphate.

This experiment should show that the finely divided metallic silver left from the decomposition of the sulphate by heat, is re-sulphatized by the sulphuric anhydride evolved from the ferrous sulphate.

Experiment 85

Sulphatizing Metallic Silver by Cupric Sulphate. — Repeat Experiment 84, substituting cupric sulphate for ferrous sulphate.

Experiment 86

The Elkington Process of Electrolizing Argentiferous Metallic Copper. — In this process anodes of argentiferous copper are electrolyzed in an electrolyte of cupric sulphate acidulated with sulphuric acid. The copper is thrown down on cathodes of metallic copper, the silver is recovered in the slimes, and all impurities in the anode copper should remain either dissolved in the electrolyte or precipitated in the slimes, so that the resultant cathode copper may be pure. The following experiments investigate the influence of the electro-motive force on (1) the rapidity of the process, (2) the consumption of power, and (3) the purity of the copper, which are the matters of chief economic importance.

Each electrolytic tank is to be a glass jar, such as are used for storage batteries, about 9 inches high, 2 inches wide and 7 inches long, with (1) as anode, a plate of copper 6 inches square and ½ inch thick, containing 3 per cent of arsenic and about 100 ounces of silver to the ton; (2) as cathode, a 6-inch square plate of copper about ⅛ inch thick; and (3) as electrolyte, water containing 3.8 per cent of copper dissolved as cupric sulphate, and 6 per cent of free sulphuric acid. The distance between anodes and cathodes is to be about 1½ inches. The current is preferably to be derived in each case from storage batteries. The outer face of both anode and cathode is to be covered with "liquid asphalt" paint, or "mounting wax," so that only the inner face of each is exposed. The inner face of the cathode should be coated with fine graphite, so that the copper deposited may readily be stripped off in order that it may be

weighed and analyzed. This should be done at the intervals indicated beyond. A stream of air should bubble through the electrolyte to prevent stratification.

In Fig. 43, A is an ammeter, V a voltmeter, B, B′, B″ are storage battery cells, T an electrolytic tank, in which *a* is the anode and *c* the cathode; U and U′ are rheostats.* The rate of deposition is to be determined in each run by weighing the deposited copper. Before weighing, the copper should be washed with water, and then with alcohol, which is immediately to be burnt off.

Four times daily, at intervals as long as practicable, record the amperage.

First Run. — Using an E. M. F. of 1½ volts (i. e., with a drop of 1½ volts from anode to cathode), make a run long enough to dissolve and re-precipitate three fourths of the copper of the anode, stripping the deposited copper and weighing it and determining its arsenic every 24 hours.

Second Run. — Using an E. M. F. of ½ volt, make a run of 2 weeks, or as much longer as is necessary to deposit approximately the same amount of copper that was deposited in the first run. Strip and weigh the deposited copper and determine its arsenic every 2 days.

Results. — A comparison of the results of the two runs should lead to the following inferences:

(1) that doubling the electro-motive force approximately doubles the amperage, as follows from Ohm's law, $Q = E.M.F. \div R$; and approximately doubles the deposition of copper. As the quantity of copper under treatment is the same in both cases, this means that the interest charges per unit of copper are inversely as the E. M. F.

(2) the watts, the measure of the energy consumed or of the cost for power, have, however, been approximately quadrupled ($W = V \times A$); indicating that the cost of the power per unit of copper deposited is proportional to the E. M. F.

Purity. — The results should show (1) that taking each run

* A form of rheostat convenient for the purpose of regulating the current for the electrolysis, is simply a soft iron wire about 19 B. W. G., four feet long, with two binding-posts, of which one can be moved along the wire. By varying the distance between these binding-posts the current resistance can be readily adjusted.

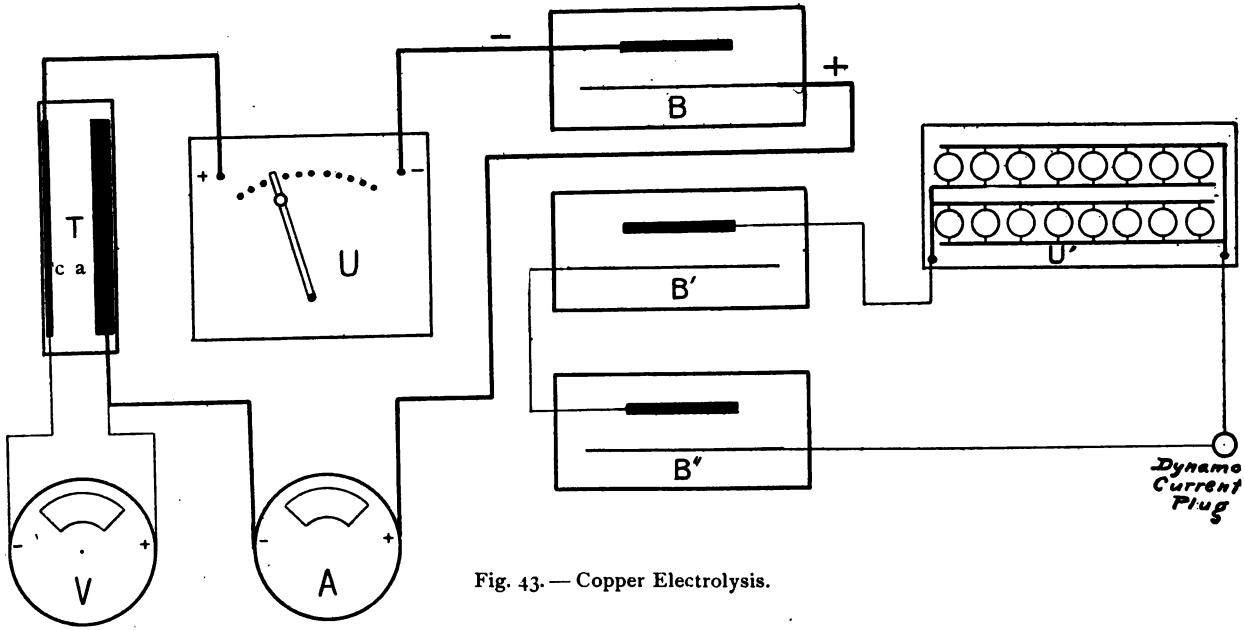


Fig. 43. — Copper Electrolysis.

Legend: A, ammeter.
B,B′,B″, battery cells.
T, electrolyzing tank:
a, anode; c, cathode.
U, rheostat regulating the current for the electrolysis.
U′, lamp-rheostat regulating the current for charging the batteries.
V, voltmeter.

Battery B only is furnishing the electrolyzing current; batteries B′ and B″ are charging by dynamo current. Thus at any given time one of the batteries is supplying the current for the electrolysis, and one or both of the two others are charging.

separately the copper first thrown down, when the electrolyte had received but little arsenic from the anode, is nearly or quite free from arsenic, but that the quantity of arsenic in the deposited copper increases as the electrolyte is enriched in arsenic.

(2) That, for given concentration of arsenic in the electrolyte, the quantity of arsenic thrown down on the cathode with the copper increases with the E. M. F.

To sum up, as the electro-motive force increases the consumption of power per unit of copper recovered increases, but the interest charges and the purity of the copper decrease. Further, as the electrolyte becomes impure, so does the copper precipitated from it.

Determine the silver precipitated in the slimes, and calculate what percentage of that dissolved from the anode this represents.

Present to the officer in charge a tabular statement of all your results, together with a discussion of the agreement between them and the inferences above stated.

Experiment 87

ALLOY DIFFUSION EXPERIMENT * OF LE CHATELIER AND STEAD. — The object of this ingenious experiment is to prepare in a single specimen a complete series of all the alloys of two given metals, by pouring a layer of one (the lighter) upon a layer of the other, giving a little time for diffusion, and then allowing the mass to solidify undisturbed. On sawing the resultant ingot open, polishing and etching the section thus obtained, the whole series of alloys may be examined under the microscope.

To avoid contaminating the metals, it will often be well to use crucibles of magnesia.

Melt 40 grammes of metallic copper under a layer of charcoal and borax, preferably in a magnesia crucible 2¼ inches high and 1⅛ inches in diameter at the top, and pour it into a second magnesia crucible of the same dimensions, and at a temperature slightly below the melting point of copper (1083° C.). To this end, this second crucible should be cautiously preheated in a small gas or electric-resistance crucible furnace to about 1050°. Without removing the crucible from the furnace, and as soon as the

* Le Chatelier, *The Metallographist,* IV, 1901, p. 17. Stead, "Metallic Alloys," Cleveland Institution of Engineers, December 10, 1900, p. 14.

lower part of the copper has solidified, pour gently upon the surface of the copper 20 grammes of aluminium previously brought to about 1050° in a third like magnesia crucible under cane-sugar, charcoal and borax. To promote diffusion, leave the crucible in the furnace for 10 minutes without lowering its temperature. Then withdraw the crucible and allow it to cool in the air.

When cold, saw the ingot in two vertically, polish the section thus exposed, and etch the lower part with concentrated hydrochloric acid, and the upper part with dilute nitric acid, adding concentrated nitric acid to it tentatively until the proper strength is reached.

If it appears that diffusion has gone too far, it can be restrained in the next trial by faster cooling, or by having the aluminium cooler when added, or even by setting a cold iron plate upon the aluminium after it has been added.

GOLD AND SILVER

Experiment 88

THE CYANIDE PROCESS.* — This is a process for extracting metallic gold and silver, but chiefly the former, from ores containing them, by means of potassium cyanide, and it is based upon the fact that when either gold or silver is exposed to the action of an aqueous solution of potassium cyanide in the presence of atmospheric oxygen, potassic auro-cyanide or argento-cyanide is formed, and, being soluble in water, is readily removed thus from the ore. The reaction is probably

$$4Au + 8KCN + 2H_2O + 2O = 4KAu(CN)_2 + 4KOH$$

In case of silver, Ag should be substituted for Au in this reaction.

The present experiment is to determine the relation between the concentration of the cyanide solution and its solvent power, taking advantage of the fact that, other things being equal, the quantity of metal dissolved is proportional to the surface exposed to the solvent.

Prepare ten solutions of 500 c.c. each, containing the following amounts of potassium cyanide: 2 each with 0.10 per cent, 0.25 per cent, 0.50 per cent, 1.0 per cent, and 2.5 per cent. Put these

* The outline of this experiment was prepared by Mr. W. A. Bentley.

solutions in litre flasks, and arrange them in pairs according to strength. Into one of each pair drop 4 square inches of gold foil; into the other drop the same area of silver foil. Label for identification, and give corresponding numbers to all vessels used subsequently.

Allow to stand five hours; agitate frequently, and take care that all the flasks receive the same treatment, and are under the same temperature conditions. At the end of this time pour the solutions through separate filters to remove any finely divided gold and silver, and catch the filtrate in beakers. Into each beaker put 2 grammes of freshly cut shavings of metallic zinc, to precipitate the gold and silver, cover, and allow to stand under the hood for at least 24 hours. Wash each precipitate once by decanting, pouring the solutions and corresponding wash-waters through filters, numbered for identification. Leave the filters in the funnels; the solutions and wash-water may be rejected.

To the washed residues add hydrochloric acid (1 part concentrated acid to 1 part water) little by little, until the effervescence caused by the dissolving zinc ceases. Pour the contents of each beaker upon the corresponding filter, wash, and while still wet transfer the filter papers with the residues to numbered scorifying cups, each containing 20 grammes of test lead. Scorify in the muffle furnace, cupel the resulting buttons, weigh the gold and silver and compare the results.

Hand your results with your own brief statement of the law to which they point, to the proper officer. In particular, cite any results which seem to conflict with this law.

Experiments 89 to 91

Chlorination Process.*—This process extracts gold from its ores by converting it by means of chlorine water into the readily soluble form of auric chloride, $AuCl_3$, which is then washed out with water. The ore, mixed with water, is exposed to chlorine gas under pressure in a lead-lined iron barrel (Fig. 44), revolving on the trunnions AB.

The carbonates and oxides of the alkaline earths, and the metallic sulphides and protosulphates interfere with the process.

* The first outline of these chlorination experiments was prepared by Mr. W. A. Bentley.

Therefore before the chlorination begins they should be removed or converted into some harmless form, as for instance by a chloridizing or oxidizing roasting. Thus pyritic ores should be roasted so thoroughly as to decompose not only the pyrites itself but also the ferrous sulphate to which it gives rise in roasting; unless it is thought cheaper to wash the ferrous sulphate out in water.

The use of this process is in general limited to ores too lean for smelting.

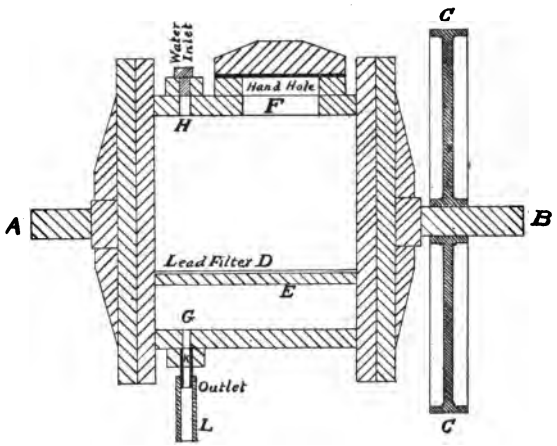


Fig. 44. — Chlorinating Barrel. — Scale 1 = 10.

Legend: *A*, *B*, trunnions.
C, pulley, driven by means of a belt.
D, perforated sheet lead filter.
E, frame to support the filter.
F, hand hole.
G, outlet.
H, water inlet.
K, threaded iron tube fitting into outlet *G*.
L, rubber tube, controlled by a pinchcock.

The following experiments address themselves to this influence of (1) pyrites, and (2) ferrous sulphate in increasing the consumption of chlorine and interfering with the extraction of the gold. To this end a first lot of ore (Experiment 89), is roasted under normal and favorable conditions: next a second and a third lot of this same roasted ore (Experiments 90 and 91), are treated while mixed with pyrites and ferrous sulphate respectively; and then the results of the three treatments are compared.

General Directions. — The Barrel has the following parts:

AB, trunnions.

C, pulley driven by means of a belt.

D, perforated sheet lead filter.

E, frame to support the filter.

F, hand hole.

G, outlet for drawing off the resultant auriferous chlorine water. This outlet is to be closed during the chlorination with a threaded iron plug, but during the subsequent leaching with a threaded iron tube K, on which is firmly fastened a rubber tube L, controlled by means of a pinchcock.

H, hole for running in water, closed by means of a threaded plug, and provided with a tight fitting for connecting the city water under its own pressure.

K, threaded iron tube fitting into the outlet G.

L, rubber tube, controlled by a pinchcock.

The Ore. — This should be a pyritic gold ore, free from the alkaline earths in such a form that after the roasting they will be acted upon by the chlorine water.

If such an ore is lacking, an artificial ore may be made by mixing finely divided gold very intimately and thoroughly with silica sand which has passed a sieve of 15 meshes to the linear inch, together with 10 per cent of iron pyrites of the same fineness.*

Roasting. — Seventy-five pounds of this ore should be roasted very thoroughly in the oven roasting furnace, until its sulphur-content does not exceed 0.30 per cent. Divide it into three equal lots, one for each of the chlorination experiments.

The Chlorinating. — A. *Charging.* — Make the barrel fast with the hand hole up, the filter level, the hand hole open, and the holes H and G closed with their plugs. Fill the barrel to within 3 inches of the top with water, and then add 25 pounds of

* The following method of preparing an artificial gold ore, though it leaves room for improvement, is offered here provisionally for lack of a better.

Preparing the gold. — Dissolve 0.8 grammes of gold in nitro-hydrochloric acid, add 30 c.c. of concentrated sulphuric acid, evaporate the solution until fumes of sulphuric acid are given off abundantly, cool, dilute, and add ferrous sulphate until all the gold is precipitated. Some care may be required to complete the precipitation of the gold. Then, by stirring, mix in 80 grammes of fine silica sand with the precipitated gold. The object of adding this sand is to facilitate mixing the gold later with the 25 pounds of sand which is to form the gangue of the ore. Unless some such precaution is taken, the precipitated gold forms

roasted and assayed ore. Take care that the part of the ore last added does not become mixed up with the water, but that its upper surface remains dry. On this, as far toward one end of the barrel as possible, put 8 ounces (2.0 per cent of the weight of the ore), of chloride of lime, called "bleaching powder." By means of a large funnel pour in toward the opposite end 16 ounces of concentrated (66° B.) sulphuric acid, and close the hand hole immediately.

B. *Chlorinating.* — Rotate the barrel about ten times a minute for four hours, then stop it with the hand hole down, remove the outlet plug from G, and allow the excess of chlorine gas to escape.

C. *Washing.* — Screw the tube K into the outlet G, close the pinch-cock on L, turn the barrel so as to bring the hand hole up, swing it gently three or four times, to settle the ore uniformly on the filter, and then fasten it with the filter level. Connecting the city water supply with the inlet H, wash the ore under city pressure until the wash water, which is removed each time through the outlet G, forms no precipitate with sulphuretted hydrogen.

Discharging. — Now discharge the ore through the hand hole, and wash the barrel clean and drain it.

Control. — Now measure the gold bearing solution, for instance in a graduated tank, stir thoroughly, allow to settle completely, and take a one-litre sample. Heat this to boiling, add 5 grammes of lead acetate dissolved in as little water as possible, and while hot pass sulphuretted hydrogen through. Filter without

---

lumps, not easy to break up into a fine powder which mixes readily with the sand, but rather tending to flatten out, weld together, and form thin flakes which do not mix well with the sand. As a consequence the gold-content of the artificial ore is very irregular, and sampling becomes very difficult.

The precipitated gold and the admixed sand are to be filtered, washed thoroughly with hot water, and dried. The filter paper is to be burned and its ashes added to the gold-bearing sand.

Making the ore. — Next mix this thoroughly with 25 pounds of quartz sand and 2.5 pounds of pyrites, each of which has been crushed so as to pass a sieve of 15 meshes to the linear inch. As the success of the experiment requires that the sampling shall be accurate, and as this in turn requires that the ore shall be extremely uniform, the mixing should be extremely careful and thorough.

washing, dry the precipitate on the paper, add about 100 milligrammes of metallic silver, and scorify the whole with 20 grammes of test lead until the lead sulphide is completely decomposed. Cupel the button, part, and weigh the gold; calculate the total amount of gold extracted by multiplying this weight by the number of litres of solution in the tank. Sample and assay the tailings. From the original assay, calculate the amount of gold in the ore treated, and, by comparing this with the amount extracted, determine the extraction percentage.

From the data at hand calculate and report the percentage of the original gold

(1) recovered, (2) left in the residue (the leached ore), (3) unaccounted for.

Experiment 89

CHLORINATING A NORMAL ORE. — Treat 25 pounds of the roasted ore exactly in the way just described.

Experiment 90

CHLORINATING IN THE PRESENCE OF RAW PYRITES. — To a second 25-pound lot of the same roasted ore, add enough iron pyrites free from gold, silver, and alkaline earths, to introduce 2 per cent of sulphur. Follow the general directions up to and through the washing of the ore after chlorination.

In Experiment 89 a smell of free chlorine will be noticed when the barrel is opened after the chlorination, showing that an excess of chlorine is present. In the present experiment, owing to the consumption of the chlorine by the pyrites present, there will probably be no decided smell of chlorine, such as there would be if an excess were present sufficient to insure thorough chloridizing.

On this account repeat the chloridizing as follows: without removing the washed ore, fill the barrel again with water to within three inches of the top, without stirring again pour in 8 ounces of chloride of lime and 16 ounces of 66° Baumé sulphuric acid, and close the barrel quickly, preventing the escape of chlorine as fully as possible. Proceed as before, and continue thus chlorinating and washing thoroughly, until on opening the barrel after the normal time for chlorinating, there is a decided smell of chlorine. Some care may be needed at first to distinguish between the smell

of hydrochloric acid, which may be present, and that of chlorine; and the student should therefore refresh his memory on this point.

The gold in the washings from each chlorination of this lot of ore should be determined separately, calculated into percentage of the total gold contents of the ore, and reported in tabular form together with the total percentage yield, the percentage of the total gold remaining in the residue, the percentage unaccounted for, and the increased consumption of sulphuric acid and chloride of lime, and also of time, necessitated by the presence of the pyrites.

Experiment 91

CHLORINATING IN THE PRESENCE OF FERROUS SULPHATE. — Chlorinate the third lot of the same roasted ore, adding to it 2 per cent of ferrous sulphate (copperas), but otherwise proceeding as in Experiment 89. Repeat the chlorinating until, on opening the barrel at the normal time, a decided smell of chlorine is noticed, showing that enough has been formed to provide for the ferrous sulphate and still to leave an excess for chlorinating the gold.

The gold in the washings after each chlorination of this lot of ore should be determined separately, calculated into percentage of the total gold contents of the ore, and reported in tabular form together with the total percentage yield, the percentage of the total gold remaining in the residue, the percentage unaccounted for, and the increased consumption of sulphuric acid and chloride of lime, and also of time, necessitated by the presence of the ferrous sulphate.

APPENDIX

Directions for Lining Crucibles

BRASQUING, I. E., LINING WITH CARBON. — Hessian or clay crucibles will generally be used for brasquing, because of their cheapness and because the brasquing does not react on them, and protects them from the charge, however corrosive. But until the student has had some experience in brasquing it is wiser to brasque graphite crucibles if corrosive matter is to be melted, so that if through defective ramming the brasquing gives way, the outer graphite crucible may prevent the corrosive materials from boring through, running out into the furnace, and in turn corroding it.

MATERIAL. — This should be finely powdered charcoal, or, in case the ash of charcoal be objectionable, then lampblack or gas carbon. The charcoal of cane-sugar is probably the best of all, though rather the most expensive.

Lampblack has the advantage of cheapness and purity, but it is so light and greasy that its use is very inconvenient. Nevertheless, the author has made some very serviceable linings with it.

The powdered carbonaceous matter should be thoroughly mixed with molasses, starch paste, or better a solution of 5 per cent of dextrine in water; and only enough of this binding material to give coherence should be used.

RAMMING. — First smear the inner surface of the crucible thoroughly with the binding material, in order to moisten the crucible itself, lest it soak up the water from the rammed lining and so prevent its adhering to the crucible walls.

Next ram the mixture of carbon and binding material in the crucible, in layers not more than ¼ inch thick, ramming each layer very firmly by means of a wooden plug about ¾ inch in diameter, and 1 foot long. Each layer, after hard ramming, is to be roughened by scratching it, so that the layer next to be rammed will adhere firmly to it. In this way fill the crucible to within ½ inch of its top.

Cut out with a knife, or better a gouge, the cavity needed,

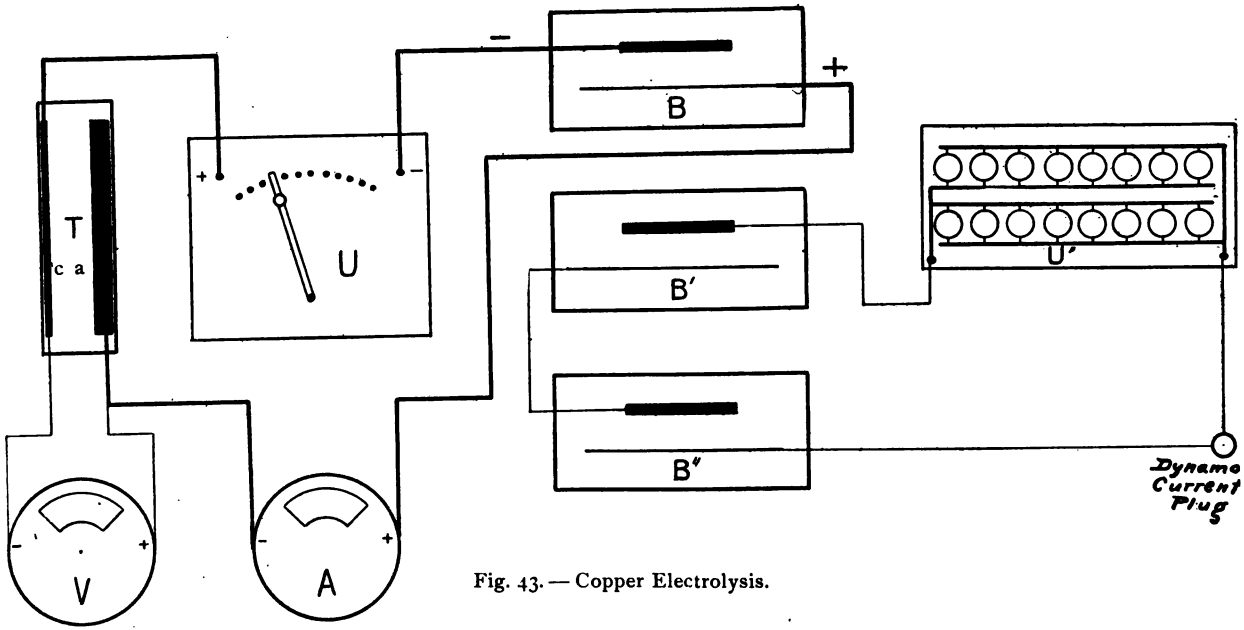


Fig. 43. — Copper Electrolysis.

Legend: A, ammeter.
B,B′,B″, battery cells.
T, electrolyzing tank:
a, anode; c, cathode.
U, rheostat regulating the current for the electrolysis.
U′, lamp-rheostat regulating the current for charging the batteries.
V, voltmeter.

Battery B only is furnishing the electrolyzing current; batteries B′ and B″ are charging by dynamo current. Thus at any given time one of the batteries is supplying the current for the electrolysis, and one or both of the two others are charging.

For industrial furnace linings, these basic and neutral materials are usually mixed with about 10 per cent of coal tar, which should be thoroughly de-hydrated by boiling, lest its water slack the lime. Tar is very inconvenient for laboratory use, because it is so sticky, but it is well that the student should use it at least once so as to know its behavior.

Excellent magnesia crucibles are made by Messrs. Muller & Co., Ivry Port, near Paris, France.

TABLES

TABLE 4. — *Atomic Weights* *

Name of Element	Atomic Weight (O=16)	Logarithm Atomic Weight	Name of Element	Atomic Weight (O=16)	Logarithm Atomic Weight
Aluminium	27.1	1.432969	Mercury	200.0	2.301030
Antimony	120.4	2.080626	Nickel	58.70	1.768638
Arsenic	75.0	1.875061	Nitrogen	14.04	1.147367
Calcium	40.1	1.603144	Oxygen	16.000	1.204120
Carbon	12.0	1.079181	Phosphorus	31.0	1.491362
Chlorine	35.45	1.549616	Platinum	194.9	2.289812
Copper	63.6	1.803457	Silicon	28.4	1.453318
Fluorine	19.05	1.279895	Silver	107.92	2.033102
Gold	197.2	2.294907	Sodium	23.05	1.362671
Hydrogen	1.008	0.003461	Sulphur	32.07	1.506099
Iron	55.9	1.747412	Tin	119.0	2.075547
Lead	206.92	2.315802	Titanium	48.15	1.682596
Magnesium	24.3	1.385606	Tungsten	184.	2.264818
Manganese	55.0	1.740363	Zinc	65.4	1.815578

* Clarke, *Jour. Am. Chem. Soc.*, XXIV, pp. 213–215, March, 1902.

TABLE 5. — *Molecular Weights, etc., of Vario*

Compound	Molecular weight of compound	Log. of molecular weight of compound	Weight of metal in compound	Log. of weight of metal in compound	Weight of metalloid in compound	Log. of weight of metalloid in compound	Compound ÷ metal	Log. of compound ÷ metal	Compound ÷ metalloid	Log. of compound ÷ metalloid
Oxides: †					O					
CaO	56.1	1.748963	40.1	1.603144	16.0	1.204120	1.399	0.145819	3.5062	0.54484
MgO	40.3	1.605305	24.3	1.385606	16.0	1.204120	1.6584	0.219699	2.5187	0.40118
(CaMg)O	48.2	1.683047	32.2	1.507856	16.0	1.204120	1.4969	0.175191	3.0125	0.47892
FeO	71.9	1.856729	55.9	1.747412	16.0	1.204120	1.2862	0.109317	4.4937	0.65260
Fe_3O_4	231.7	2.364926	167.7	2.224533	64.0	1.806180	1.3816	0.140393	3.6203	0.55874
Fe_2O_3	159.8	2.203577	111.8	2.048442	48.0	1.681241	1.4293	0.155135	3.32917	0.52233
CO	28.0	1.447158	12.0	1.079181	16.0	1.204120	2.3333	0.367977	1.750	0.24303
CO_2	44.0	1.643453	12.0	1.079181	32.0	1.505150	3.6666	0.564272	1.375	0.13830
SiO_2	60.4	1.781037	28.4	1.453318	32.0	1.505150	2.1267	0.327719	1.8875	0.27588
Al_2O_3	102.2	2.009451	54.2	1.733999	48.0	1.681241	1.8856	0.275452	2.1291	0.32821
P_2O_5	142.0	2.152288	62.0	1.792392	80.0	1.903090	2.2903	0.359896	1.775	0.24919
Sulphides:					S					
Cu_2S	159.27	2.202133	127.2	2.104487	32.07	1.506099	1.2521	0.097646	4.9663	0.69603
FeS	87.97	1.944335	55.9	1.747412	32.07	1.506099	1.5737	0.196923	2.74306	0.43823
FeS_2	120.04	2.079325	55.9	1.747412	64.14	1.807129	2.1474	0.331913	1.8715	0.27219
PbS	238.99	2.378380	206.92	2.315802	32.07	1.506099	1.1549	0.062578	7.4521	0.87228
Carbonates:					C					
$FeCO_3$	115.9	2.064083	55.9	1.747412	12.0	1.079181	2.0733	0.316671	9.6583	0.98490
$CaCO_3$	100.1	2.000434	40.1	1.603144	12.0	1.079181	2.4962	0.397290	8.34168	0.92125
$MgCO_3$	84.3	1.925828	24.3	1.385606	12.0	1.079181	3.4691	0.540222	7.0250	0.84664
$(CaMg)CO_3$	92.2	1.964731	32.2	1.507856	12.0	1.079181	2.8633	0.456875	7.6833	0.88555
Silicates:					Si					
$Ca_2Si_3O_8$	293.4	2.467460	80.2	1.904174	85.2	1.930440	3.6583	0.563286	3.4436	0.53702
$CaSiO_3$	116.5	2.066326	40.1	1.603144	28.4	1.453318	2.9052	0.463182	4.1021	0.61300
$Ca_4Si_3O_{10}$	405.6	2.608098	160.4	2.205204	85.2	1.930440	2.52868	0.402894	4.7605	0.67765
Ca_2SiO_4	172.6	2.237041	80.2	1.904174	28.4	1.453318	2.1521	0.332867	6.0774	0.78372
Ca_3SiO_5	228.7	2.359266	120.3	2.080266	28.4	1.453318	1.9010	0.279000	8.0528	0.90594
Ca_4SiO_6	284.8	2.454540	160.4	2.2052c4	28.4	1.453318	1.7755	0.249336	10.0281	1.00122
$Mg_2Si_3O_8$	261.8	2.417970	48.6	1.686636	85.2	1.930440	5.3868	0.731334	3.0727	0.48753
$MgSiO_3$	100.7	2.003029	24.3	1.385606	28.4	1.453318	4.1440	0.617423	3.5457	0.54971

rganic Compounds and their Percentage Composition

Oxygen ÷ metal	Log. of oxygen ÷ metal	Metalloid ÷ metal	Log. of metalloid ÷ metal	Metal ÷ metalloid	Log. of metal ÷ metalloid	Percentage composition: Metal	Base	Acid	Oxygen or Sulphur	Compound
									O	*Oxides:* †
990	$\bar{1}$.600976	—	—	2.5062	0.399024	71.48	—	—	28.52	CaO
5843	$\bar{1}$.818514	—	—	1.5187	0.181486	60.30	—	—	39.70	MgO
9689	$\bar{1}$.696264	—	—	2.0125	0.303736	66.80	—	—	33.19	(CaMg)O
3622	$\bar{1}$.456708	—	—	3.4937	0.543292	77.75	—	—	22.25	FeO
3163	$\bar{1}$.581647	—	—	2.6203	0.418353	72.378	—	—	27.622	Fe_3O_4
2933	$\bar{1}$.632799	—	—	2.3291	0.367201	69.962	—	—	30.038	Fe_2O_3
333	0.124939	—	—	0.7500	$\bar{1}$.875061	42.857	—	—	57.143	CO
566	0.425969	—	—	0.3750	$\bar{1}$.574031	27.272	—	—	72.728	CO_2
267	0.051832	—	—	0.8875	$\bar{1}$.948168	47.019	—	—	52.981	SiO_2
85608	$\bar{1}$.947242	—	—	1.1291	0.052758	53.033	—	—	46.967	Al_2O_3
903	0.110698	—	—	0.7750	$\bar{1}$.889302	43.662	—	—	56.338	P_2O_5
									S	*Sulphides:*
—	—	0.25212	$\bar{1}$.401612	3.9663	0.598388	79.864	—	—	20.136	Cu_2S
—	—	0.573702	$\bar{1}$.758687	1.74306	0.241313	63.544	—	—	36.456	FeS
—	—	1.1474	0.059717	0.87153	$\bar{1}$.940283	46.568	—	—	53.432	FeS_2
—	—	0.15498	$\bar{1}$.190297	6.4521	0.809703	86.581	—	—	13.419	PbS
								CO_2		*Carbonates:*
35867	$\bar{1}$.933829	0.2146	$\bar{1}$.331769	4.6583	0.668231	48.231	62.036	37.964	—	$FeCO_3$
9708	0.078097	0.2992	$\bar{1}$.476037	3.3416	0.523963	40.06	56.044	43.956	—	$CaCO_3$
07531	0.295635	0.4938	$\bar{1}$.693575	2.0250	0.306425	28.825	47.805	52.194	—	$MgCO_3$
1906	0.173385	0.3726	$\bar{1}$.571325	2.6833	0.428675	34.92	52.28	47.72	—	$(CaMg)CO_3$
								SiO_2		*Silicates:*
5960	0.203036	1.0623	0.026266	0.9413	$\bar{1}$.973734	27.33	38.24	61.76	—	$Ca_2Si_3O_8$
1970	0.078097	0.7082	$\bar{1}$.850174	1.4119	0.149826	34.42	48.27*	51.72*	—	$CaSiO_3$
997507	$\bar{1}$.998916	0.5311	$\bar{1}$.725236	1.8826	0.274764	39.55	55.46*	44.53*	—	$Ca_4Si_3O_{10}$
798005	$\bar{1}$.902006	0.3541	$\bar{1}$.549144	2.8239	0.450856	46.47	65.17*	34.82*	—	Ca_2SiO_4
665003	$\bar{1}$.822824	0.2360	$\bar{1}$.373052	4.2359	0.626948	52.60	73.68*	26.31*	—	Ca_3SiO_5
598504	$\bar{1}$.777067	0.1770	$\bar{1}$.248114	5.6479	0.751886	56.32	78.88*	21.11*	—	Ca_4SiO_6
6337	0.420574	1.7530	0.243804	0.5704	$\bar{1}$.756196	18.56	30.79	69.21	—	$Mg_2Si_3O_8$
9753	0.295635	1.1687	0.067712	0.8556	$\bar{1}$.932288	24.13	40.02	59.98	—	$MgSiO_3$

TABLE 5 (*continued*). — *Molecular Weights, etc., of Vari*

Compound	Molecular weight of compound	Log. of molecular weight of compound	Weight of metal in compound	Log. of weight of metal in compound.	Weight of metalloid in compound	Log. of weight of metalloid in compound	Compound ÷ metal	Log. of compound ÷ metal	Compound ÷ metalloid	Log. of compound ÷ metalloid
Silicates:										
$Mg_4Si_3O_{10}$	342.4	2.534534	97.2	1.987666	85.2	1.930440	3.5226	0.546868	4.0187	0.6040
Mg_2SiO_4	141.0	2.149219	48.6	1.686636	28.4	1.453318	2.9012	0.462583	4.9648	0.6959
Mg_3SiO_5	181.3	2.258398	72.9	1.862728	28.4	1.453318	2.48696	0.395670	6.3838	0.8050
Mg_4SiO_6	221.6	2.345570	97.2	1.987666	28.4	1.453318	2.2798	0.357904	7.8028	0.8922
$Fe_2Si_3O_8$	325.0	2.511883	111.8	2.048442	85.2	1.930440	2.90697	0.463441	3.8145	0.5814
$FeSiO_3$	132.3	2.121560	55.9	1.747412	28.4	1.453318	2.3667	0.374148	4.6584	0.6682
$Fe_4Si_3O_{10}$	468.8	2.670988	223.6	2.349472	85.2	1.930440	2.0966	0.321516	5.5023	0.7405
Fe_2SiO_4	204.2	2.310056	111.8	2.048442	28.4	1.453318	1.82647	0.261614	7.1901	0.8567
Fe_3SiO_5	276.1	2.441066	167.7	2.224533	28.4	1.453318	1.64638	0.216533	9.7218	0.9877
Fe_4SiO_6	348.0	2.541579	223.6	2.349472	28.4	1.453318	1.5563	0.192107	12.2535	1.0882
Phosphates:					P					
CaP_2O_6	198.1	2.296884	40.1	1.603144	62.0	1.792392	4.94014	0.693740	3.1951	0.5044
$Ca_2P_2O_7$	254.2	2.405176	80.2	1.904174	62.0	1.792392	3.1696	0.501002	4.1000	0.6127
$Ca_5P_4O_{15}$	564.5	2.751664	200.5	2.302114	124.0	2.093422	2.8155	0.449550	4.5524	0.6582
$Ca_3P_2O_8$	310.3	2.491782	120.3	2.080266	62.0	1.792392	2.5794	0.411516	5.0048	0.6993
$Ca_{10}P_6O_{25}$	987.0	2.994317	401.0	2.603144	186.0	2.269513	2.4613	0.391173	5.3064	0.7248
$Ca_4P_2O_9$	366.4	2.563955	160.4	2.205204	62.0	1.792392	2.2843	0.358751	5.9096	0.7715
$Ca_5P_2O_{10}$	422.5	2.625827	200.5	2.302114	62.0	1.792392	2.1072	0.323713	6.8145	0.8334
FeP_2O_6	213.9	2.330211	55.9	1.747412	62.0	1.792392	3.8265	0.582799	3.4500	0.5378
$Fe_2P_2O_7$	285.8	2.456c62	111.8	2.048442	62.0	1.792392	2.5563	0.407620	4.6096	0.6636
$Fe_5P_4O_{15}$	643.5	2.808549	279.5	2.446382	124.0	2.093422	2.3023	0.362167	5.1895	0.7151
$Fe_3P_2O_8$	357.7	2.553519	167.7	2.224533	62.0	1.792392	2.1330	0.328986	5.7693	0.7611
$Fe_{10}P_6O_{25}$	1145	3.058805	559.0	2.747412	186.0	2.269513	2.0483	0.311393	6.1559	0.7892
$Fe_4P_2O_9$	429.6	2.633064	223.6	2.349472	62.0	1.792392	1.9213	0.283592	6.9290	0.8406
$Fe_5P_2O_{10}$	501.5	2.700271	279.5	2.446382	62.0	1.792392	1.7943	0.253889	8.0887	0.9078

* The atomic weights used to calculate this percent

† In this group (Oxides), Carbon, Silicon, and Phosphc

ganic Compounds and their Percentage Composition

	Log. of oxygen ÷ metal	Metalloid ÷ metal	Log. of metalloid ÷ metal	Metal ÷ metalloid	Log. of metal ÷ metalloid	Percentage composition				Compound
						Metal	Base	Acid	Oxygen or Sulphur	
										Silicates:
609	0.216454	0.8765	$\bar{1}$.942774	1.1408	0.057226	28.39	47.08	52.92	—	$Mg_4Si_3O_{10}$
687	0.119544	0.5843	$\bar{1}$.766682	1.7112	0.233318	34.47	57.16	42.84	—	Mg_2SiO_4
739	0.040362	0.3895	$\bar{1}$ 590590	2.5669	0.409410	40.21	66.69	33.31	—	Mg_3SiO_5
765	$\bar{1}$.994605	0.2921	$\bar{1}$.465652	3.4225	0.534348	43.86	72.74	27.26	—	Mg_4SiO_6
49	0.058768	0.7620	$\bar{1}$.881998	1.3122	0.118002	34.40	44.25	55.75	--	$Fe_2Si_3O_8$
867	$\bar{1}$.933829	0.5080	$\bar{1}$.705906	1.9683	0.294094	42.25	54.54*	45.45*	—	$FeSiO_3$
556	$\bar{1}$.854648	0.3810	$\bar{1}$.580968	2.6244	0.419032	47.70	61.55*	38.44*	—	$Fe_4Si_3O_{10}$
245	$\bar{1}$.757738	0.2540	$\bar{1}$.404876	3.9366	0.595124	54.75	70.64*	29.35*	—	Fe_2SiO_4
704	$\bar{1}$.678557	0.1693	$\bar{1}$.228785	5.9049	0.771215	60.74	78.26*	21.73*	—	Fe_3SiO_5
9337	$\bar{1}$.632799	0.1270	$\bar{1}$.103846	7.8732	0.896154	64.25	82.76*	17.23*	—	Fe_4SiO_6
								P_2O_5		*Phosphates:*
940	0.379127	1.5461	0.189248	0.6467	$\bar{1}$.810752	20.24	28.29*	71.70*	—	CaP_2O_6
865	0.145044	0.7730	$\bar{1}$.888218	1.2935	0.111782	31.55	44.08*	55.91*	—	$Ca_2P_2O_7$
970	0.078097	0.6184	$\bar{1}$.791308	1.6169	0.208692	35.52	49.64*	50.35*	—	$Ca_5P_4O_{15}$
640	0.026944	0.5153	$\bar{1}$.712126	1.9403	0.287874	38.77	54.19*	45.81*	—	$Ca_3P_2O_8$
9751	$\bar{1}$.998916	0.4638	$\bar{1}$.666369	2.1559	0.333631	40.63	56.81*	43.18*	—	$Ca_{10}P_6O_{25}$
9776	$\bar{1}$.953158	0.3865	$\bar{1}$.587188	2.5871	0.412812	43.78	61.18*	38.81*	—	$Ca_4P_2O_9$
9801	$\bar{1}$.902006	0.3092	$\bar{1}$.490278	3.2338	0.509722	47.46	66.35*	33.64*	—	$Ca_5P_2O_{10}$
174	0.234859	1.1091	0.044980	0.9016	$\bar{1}$.955020	26.13	33.65*	66.34*	—	FeP_2O_6
018	0.000776	0.5545	$\bar{1}$.743950	1.8032	0.256050	39.12	50.35*	49.65*	—	$Fe_2P_2O_7$
5868	$\bar{1}$.933829	0.4436	$\bar{1}$.647040	2.2540	0.352960	43.43	55.90*	44.09*	—	$Fe_5P_4O_{15}$
6327	$\bar{1}$.882677	0.3697	$\bar{1}$.567859	2.7048	0.432141	46.88	60.56*	39.44*	—	$Fe_3P_2O_8$
15563	$\bar{1}$.854648	0.3327	$\bar{1}$.522101	3.0053	0.477899	48.82	62.84*	37.15*	—	$Fe_{10}P_6O_{25}$
4401	$\bar{1}$.808890	0.2772	$\bar{1}$.442920	3.6064	0.557080	52.05	66.96*	33.03*	—	$Fe_4P_2O_9$
7245	$\bar{1}$.757738	0.2218	$\bar{1}$.346010	4.5080	0.653990	55.73	71.71*	28.28*	—	$Fe_5P_2O_{10}$

position were slightly different from those given in Table 9.
classed in the table as metals; Oxygen, as the metalloid.

TABLE 6. — *Synoptical Table for Microscop*

O. refers to Osmond's "Méthode Générale pour l'Analyse Micrographiqu
R. A. 5 refers to Roberts-Austen, 5th Report Allo
Mr. A. Sauveur has suggeste

	Etching-Polishing (Polissage attaque)		Under Iodine	HNO_3	Form	Bas-relief Polishing	Occurrenc
	With licorice	With NH_4NO_3					
Ferrite . . .	Colorless	Grains show heterogeneousness	Colorless	Marks the joints, colors the grains very capriciously (O., p. 13) very dilute, develops structure	Polygonal	Softest of all	In all unhardened hypoeutectic steel
Cementite	White	Not acted on	Colorless, white (R. A. 5, p. 54)	20% does not affect it in 40 secs. (O., p. 15)	In blister steel large straight lamellæ juxtaposed; polygon network of groups of lamellæ; and simple lamellæ usually curved interstratified with ferrite	Hardest of all	In all unhardened hypereutectic steel
Martensite	Colors very little, yellowish at most (O., pp. 18, 19)	Its needles appear (the sharper the less C.)	Colors but slowly (p. 18), deepness increasing with C.	Colors yellowish	Straight fibres, parallel, crossing each other often (O., p. 20)	Harder than ferrite and troostite (O., p. 19); decreases as its C. decreases with rising T. max.	Quenched steel, both high and low carbo (O., p. 18
Troostite .	Colored	Yellow brown or blue bands merging	Colors faster than martensite	Colors darker than martensite	Nearly amorphous to lightly granular and mammillary (O., p. 19)	Softer than martensite, harder than ferrite (O., p. 19)	Steel quenched quickly in A_3-A_1 rang or slowly quenched from above that range (O., p. 18)
Sorbite . .	Colored	Colored	Colors fastest; more uniformly than troostite	Colors dark			Steel slowly quenched in A_3-A_1 range
Pearlite . .	Colored	Components etch to unequal depths	Colors dark (R. A. 5, p. 54)	Colors dark	Lamellæ usually curved, never crossing each other (O , p. 20)	Harder than ferrite	In all unhardened steel
Austenite.	Colorless	Not acted on. See Metall. III, 1	Colors	Colors lighter than martensite		Softer than martensite	In high carbon steel, very suddenly cooled

Aciers au Carbone," *Bulletin de la Société d'Encouragement*, May 1895.
search Committee. — Metall. refers to *The Metallographist*.
eral of these entries.

ccom-anies	Transition	Lustre	Composition	Differs	Notes
arlite		Splendent	Iron, nearly pure	from all by softness (O., p. 20.) In hard steels from cementite, colored by nitric acid (O., p. 20)	
arlite		Splendent silver-white under direct illumination (O., p. 15)	Fe_3C	from all, in hardness (O., p. 20); from ferrite, in hard steels, by not being colored by nitric acid (O., p. 20)	As it decomposes on rising above Ac_1, it passes through atmosphere of sorbite, into martensite, which has to encroach on ferrite (because, being leaner in carbon than cementite, it must take iron from the ferrite); but between martensite and ferrite is zone of troostite.
rite in y low rbon teel, entite in y high rbon teel	Austenite to troostite (O., p. 21)	Pearly under oblique light	Crystalline organization of some allotropic varieties of iron under influence of C. (O., p. 18) varies,—richer in Fe as T. max. increases because % of martensite increases with T. max. (conditions of carbon in iron)	from pearlite, in having straight fibres which intersect each other (O., p. 20)	Crystals become larger and more regularly grouped, with planes of low cohesion along edges of groups (O., p. 30), as T. max. rises.
rten-ite	insensibly, as carbon into each other	Martensite to sorbite and pearlite	Contains less hardening carbon than martensite, but more than sorbite (different allotropic modifications of solidified solutions)	from sorbite; colors more slowly and less uniformly than sorbite, and accompanies martensite (O., p. 20)	Can be formed in steel containing 0.40 per cent or less of carbon by quenching it from about 750° C. (1382° F. R. A., p. 56).
arlite	Pass into ferrite diminishes pass	Troostite to pearlite	Contains carbon because iodine colors it; probably hardening carbon (O., p. 17)	from troostite; colors faster and more uniformly, accompanies pearlite (O., p. 20)	
rite in ypo-tectic teel, entite hyper-tectic teel		Pearly under oblique light (O., p. 15)	Contains about 0.80% C; is a conglomerate of 12% of cementite with 88% of ferrite	from martensite; fibres usually curved, never cross each other (O., p. 20)	If much manganese is present, occupies abnormally large space, and is much finer.
rten-ite				from martensite; in 10% ammonium chloride solution and bichromate cell martensite colors yellow, then brown, then black (Metall. III, 3)	

of hydrochloric acid, which may be present, and that of chlorine; and the student should therefore refresh his memory on this point.

The gold in the washings from each chlorination of this lot of ore should be determined separately, calculated into percentage of the total gold contents of the ore, and reported in tabular form together with the total percentage yield, the percentage of the total gold remaining in the residue, the percentage unaccounted for, and the increased consumption of sulphuric acid and chloride of lime, and also of time, necessitated by the presence of the pyrites.

Experiment 91

CHLORINATING IN THE PRESENCE OF FERROUS SULPHATE. — Chlorinate the third lot of the same roasted ore, adding to it 2 per cent of ferrous sulphate (copperas), but otherwise proceeding as in Experiment 89. Repeat the chlorinating until, on opening the barrel at the normal time, a decided smell of chlorine is noticed, showing that enough has been formed to provide for the ferrous sulphate and still to leave an excess for chlorinating the gold.

The gold in the washings after each chlorination of this lot of ore should be determined separately, calculated into percentage of the total gold contents of the ore, and reported in tabular form together with the total percentage yield, the percentage of the total gold remaining in the residue, the percentage unaccounted for, and the increased consumption of sulphuric acid and chloride of lime, and also of time, necessitated by the presence of the ferrous sulphate.

APPENDIX

Directions for Lining Crucibles

BRASQUING, I. E., LINING WITH CARBON. — Hessian or clay crucibles will generally be used for brasquing, because of their cheapness and because the brasquing does not react on them, and protects them from the charge, however corrosive. But until the student has had some experience in brasquing it is wiser to brasque graphite crucibles if corrosive matter is to be melted, so that if through defective ramming the brasquing gives way, the outer graphite crucible may prevent the corrosive materials from boring through, running out into the furnace, and in turn corroding it.

MATERIAL. — This should be finely powdered charcoal, or, in case the ash of charcoal be objectionable, then lampblack or gas carbon. The charcoal of cane-sugar is probably the best of all, though rather the most expensive.

Lampblack has the advantage of cheapness and purity, but it is so light and greasy that its use is very inconvenient. Nevertheless, the author has made some very serviceable linings with it.

The powdered carbonaceous matter should be thoroughly mixed with molasses, starch paste, or better a solution of 5 per cent of dextrine in water; and only enough of this binding material to give coherence should be used.

RAMMING. — First smear the inner surface of the crucible thoroughly with the binding material, in order to moisten the crucible itself, lest it soak up the water from the rammed lining and so prevent its adhering to the crucible walls.

Next ram the mixture of carbon and binding material in the crucible, in layers not more than ¼ inch thick, ramming each layer very firmly by means of a wooden plug about ¾ inch in diameter, and 1 foot long. Each layer, after hard ramming, is to be roughened by scratching it, so that the layer next to be rammed will adhere firmly to it. In this way fill the crucible to within ½ inch of its top.

Cut out with a knife, or better a gouge, the cavity needed,

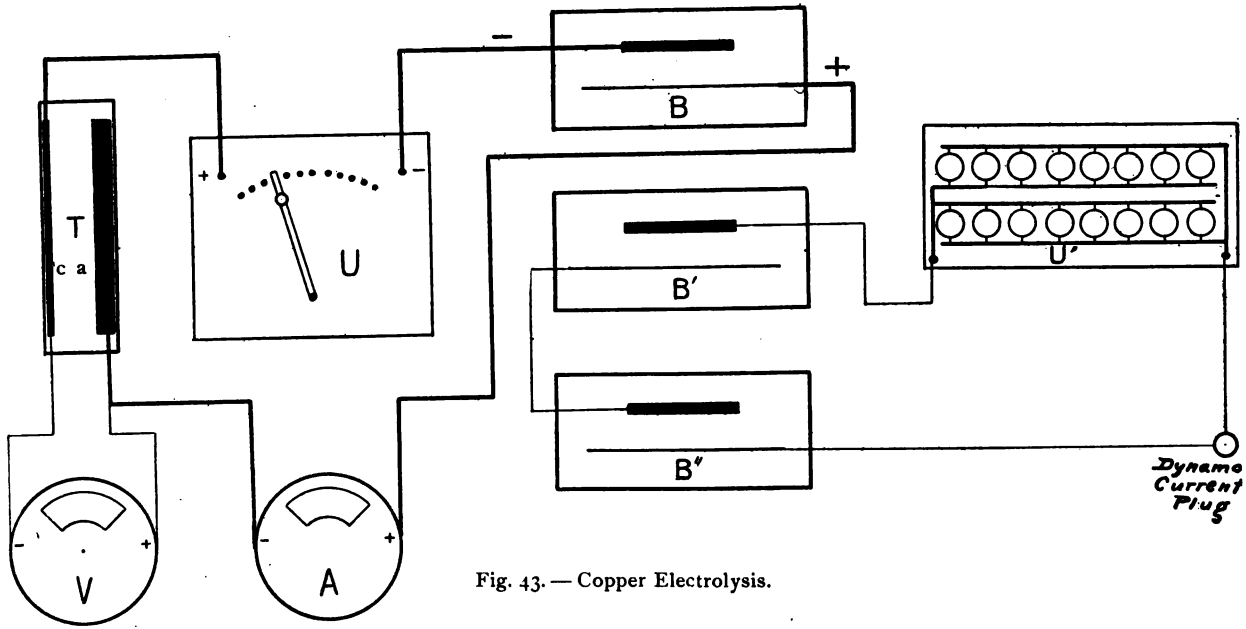


Fig. 43. — Copper Electrolysis.

Legend: A, ammeter.
B,B′,B″, battery cells.
T, electrolyzing tank:
a, anode; c, cathode.
U, rheostat regulating the current for the electrolysis.
U′, lamp-rheostat regulating the current for charging the batteries.
V, voltmeter.

Battery B only is furnishing the electrolyzing current; batteries B′ and B″ are charging by dynamo current. Thus at any given time one of the batteries is supplying the current for the electrolysis, and one or both of the two others are charging.

separately the copper first thrown down, when the electrolyte had received but little arsenic from the anode, is nearly or quite free from arsenic, but that the quantity of arsenic in the deposited copper increases as the electrolyte is enriched in arsenic.

(2) That, for given concentration of arsenic in the electrolyte, the quantity of arsenic thrown down on the cathode with the copper increases with the E. M. F.

To sum up, as the electro-motive force increases the consumption of power per unit of copper recovered increases, but the interest charges and the purity of the copper decrease. Further, as the electrolyte becomes impure, so does the copper precipitated from it.

Determine the silver precipitated in the slimes, and calculate what percentage of that dissolved from the anode this represents.

Present to the officer in charge a tabular statement of all your results, together with a discussion of the agreement between them and the inferences above stated.

Experiment 87

ALLOY DIFFUSION EXPERIMENT * OF LE CHATELIER AND STEAD. — The object of this ingenious experiment is to prepare in a single specimen a complete series of all the alloys of two given metals, by pouring a layer of one (the lighter) upon a layer of the other, giving a little time for diffusion, and then allowing the mass to solidify undisturbed. On sawing the resultant ingot open, polishing and etching the section thus obtained, the whole series of alloys may be examined under the microscope.

To avoid contaminating the metals, it will often be well to use crucibles of magnesia.

Melt 40 grammes of metallic copper under a layer of charcoal and borax, preferably in a magnesia crucible 2¼ inches high and 1⅛ inches in diameter at the top, and pour it into a second magnesia crucible of the same dimensions, and at a temperature slightly below the melting point of copper (1083° C.). To this end, this second crucible should be cautiously preheated in a small gas or electric-resistance crucible furnace to about 1050°. Without removing the crucible from the furnace, and as soon as the

* Le Chatelier, *The Metallographist,* IV, 1901, p. 17. Stead, "Metallic Alloys," Cleveland Institution of Engineers, December 10, 1900, p. 14.

TABLE 10. — *List of the Chief Special Apparatus Needed for the Experiments of this Book*

Description	See Fig. No.	Used in Experiments
Le Chatelier Thermo-Electric Pyrometer	1 and 2	1, 6, 11, 15 to 45, 47, 57 to 64, 66 to 69, 73 to 82, 84, and 85
Optical Pyrometers of Le Chatelier and Mesuré and Nouel	—	15
Siemens' Water Pyrometer	—	11
Mahler's Berthelot Calorimeter	11	12
Polishing Machine	—	5, 57, 58, 61, 63 to 69
Microscope for Examining Opaque Objects	—	5, 57, 58, 61, 63 to 69, 81, 82
Drop-Testing Machine	8	4, 48, 49, 59, 60, 62
Tensile Testing Machine for Wire*	10	8, 9, 56
Mould for Making Silicate Pyramids	17 and 18	21 to 38
Cast Iron Ingot Mould	28	49
Fire Brick " "	29	49
Electric-Resistance Crucible Furnace	13	13, 47, 81, 82, 84, 85
Small Square Gas Forge†	19 and 20	3, 21 to 38
Cylindrical " " †	36 and 37	4, 11, 57 to 64
Gas Oven Furnace†	9	6, 73 to 80, 89 to 91
Gas Crucible Furnace†	26	40 to 45
Small Muffle for use in the Square Gas Forge	19 and 20	21 to 38
" " " " Cylindrical Gas Forge	36 and 37	57, 59, 61 to 64
Desilverizing Kettle	—	7
Storage Battery Cells	43	86
Electrolytic Outfit	43	86
Chlorinating Barrel	44	89 to 91

* A tensile testing machine of the usual type, although very useful, is not essential to this work.

† These four furnaces are made by the American Gas Furnace Company, New York.

INDEX

INDEX

A

PAGE

Acid, nitric, correction for formation of..... 36
Acid, nitric, heat of formation of..... 35
Acid, nitric, quantitative determination of..... 35
Acid, sulphuric, quantitative determination of..... 36
Acidimetry 35
Acidity, its effect on melting point of lead silicates..... 59
Alloy, composition of Levol's..... 18
Alloy diffusion experiment..... 107
Alloys, copper-aluminium, diffusion of..... 107
Alloys, copper-silver, metallography..... 17
Alumina-lime silicates, melting point determinations of.....55, 57
Alumina-lime silicates, melting points of..... 126
Aluminium-copper alloys, diffusion of..... 107
Aluminium melting point determination..... 9
Analysis of roasted copper sulphide ore..... 22
Apparatus required, table of special..... 128
Artificial gold ore, preparation of..... 111
Assay (see Analysis).
Atomic weights, table of..... 117
Austenite 122, 123

B

Barrel for chlorination..... 110
Base bullion, desilverization of..... 24
Basic linings, preparation of..... 116
Basic refractory materials, slacking of..... 62
Berthelot calorimeter.....31-36
Binding material for silicates..... 46
Binding materials for crucible linings..... 116
Blister steel, microstructure of.....88, 89
Boiler plate steel, finishing temperature for..... 88
Boiling points, table of.....124, 125
Brasquing crucibles..... 115
Brinell's tests on fracture of steel.....75-78

PAGE

Brittleness (see Ductility).
Burning hypo-eutectic steel.....82, 83
Burning of steel, rationale of..... 82

C

Calibration of Le Chatelier thermo-electric pyrometer.....3, 5-8, 9, 38, 39
Calorific power, determination of.....31-36
Calorific power of iron..... 36
Calorimeter, Mahler's Berthelot.....31-36
Calorimetry and pyrometry, experiments on.....30-46
Calorimetry, cooling correction for..... 35
Carbon-content, its relation to hardening power of steel..... 29
Carbon crucibles..... 116
Carbonates, molecular weights, percentage compositions, etc., of..118, 119
Cast iron, fracture of..... 73
Cementite 122, 123
Charcoal, lining crucibles with..... 115
Chloridizing roasting.....94-98
Chlorinating barrel..... 110
Chlorination process..... 109
Chromite, corrosion of, by silicates.....62-69
Chromite linings, preparation of..... 116
Coal tar for binding materials, use of..... 117
Cold junction of Le Chatelier pyrometer..... 5
Cold working, effects of (see Overstrain).
Color temperatures.....124, 125
Colors, temperatures of temper..... 124
Comparison of thermometric scales, table of..... 127
Composition of Levol's alloy..... 18
Composition of Muntz metal..... 92
Composition, percentage, of inorganic compounds, table of.....118-121
Constitution of copper-silver alloys..... 17
Constitutional changes in steel during refining.....80-82
Contraction of refractory materials.....60-62
Converting temperatures, formulæ for..... 127
Cooling correction for calorimetry..... 35
Copper-aluminium alloys, diffusion of..... 107
Copper electrolysis.....104-107
Copper, experiments on.....91-108
Copper, its relative affinity for oxygen and sulphur..... 93
Copper melting point determination..... 38
Copper ore, chloridizing roasting of.....94-98
Copper selecting process..... 91
Copper-silver alloys, metallography of..... 17
Copper, spelter test for purity of..... 92
Copper, sulphatization of.....101, 102

PAGE
Copper sulphide ore, analysis of roasted..... 22
Copper sulphide ore, oxidizing roasting of.....19-24
Copper, Ziervogel process.....98-104
Correction, cooling, for calorimetry..... 35
Correction for cold junction, Le Chatelier pyrometer..... 5
Correction for formation of nitric acid..... 35
Corrections, Mahler's Berthelot calorimeter.....35, 36
Corrosion of refractory materials by silicates.....62-69
Cost calculation for chloridizing roasting..... 97
Critical point of 0.8 carbon steel, determination of..... 8
Critical points of steel.....70, 71, 72, 78, 85, 124, 125
Crucibles, carbon..... 116
Crucibles, lining of..... 115
Crucibles, magnesia..... 117
Cupric sulphate, sulphatizing action of.....102, 104
Cyanide process..... 108

D

Desilverization of base bullion..... 24
Desulphurization of iron by manganese..... 69
Determination of nitric acid, quantitative..... 35
Determination of sulphuric acid, quantitative..... 36
Dextrine water for binding material, use of..... 115
Diffusion of copper-aluminium alloys..... 107
Dolomite linings, preparation of..... 116
Drop-tests, directions for making..... 14
Ductility of steel, effect of heat-treatment on.....11, 12, 15, 16, 73

E

Economic conditions of chloridizing roasting..... 97
Economic conditions of copper electrolysis..... 105
Electric-resistance furnace, directions for using..... 87
Electrolysis of copper (Elkington process).....104-107
Embrittling of steel by overheating..... 14
E. M. F., its influence in electrolysis of copper.....105, 107
Etching (hypo-eutectic steel) directions for..... 18

F

Ferric oxide, its influence in chloridizing roasting..... 98
Ferric oxide, its influence on melting points of lead silicates..... 60
Ferrite 122, 123
Ferrous sulphate, its effect in chlorination..... 114

PAGE
Ferrous sulphate, sulphatizing action of....101, 103
Ferrous sulphide, sulphatizing action of.... 102
Finishing temperature....84-88
Fire-clay, corrosion of, by silicates....62-69
Fluorine, its influence on fusibility of earthy silicates.... 53
Formation point of silicates....56-59
Formulæ for converting temperatures.... 127
Fracture of cast iron, effect of heat-treatment on.... 73
Fracture of Muntz metal.... 92
Fracture of steel, effect of heat-treatment on....12, 13, 16, 75-78, 90
Furnace, cylindrical gas forge....80, 81
Furnace, electric-resistance crucible.... 37
Furnace, electric-resistance cylinder, directions for using.... 87
Furnace, gas crucible.... 66
Furnace, gas oven.... 20
Furnace, special muffle, for silicate melting point determinations....50-53
Furnace, square gas forge....48, 49
Furnaces, relative uniformity of gas and direct-fired.... 45
Fusibility of silicates (slags), experiments on (see Melting points) 46-60

G

Gas and direct-fired furnaces, relative uniformity of.... 45
Gold and silver, experiments on....108-114
Gold, chlorination process for.... 109
Gold, cyanide process for.... 108
Gold ore, preparation of artificial.... 111
Gradient, thermal, determinations....41-44

H

Hardening power of steel, its relation to temperature....10-12, 27-29
Hardening power of steel, its relation to carbon-content.... 29
Hardening power of steel, its relation to magnetic properties.... 12
Hardening temperature of carbon steel....11, 12
Hardness, removal of....27-29
Heat of formation of nitric acid.... 35
Heat-treatment, its effect on ductility of steel....11, 12, 15, 16, 73
Heat-treatment, its effect on fracture of cast iron.... 73
Heat-treatment, its effect on fracture of steel....13, 16, 75-78, 81, 82
Heat-treatment, its effect on microstructure of steel....80-82
Heat-treatment, its effect on overstrain....29, 79
Heat-treatment, tempering of steel.... 27
Hessian crucibles, corrosion of, by silicates....62-69
Hypo-eutectic steel, burning of....82, 83
Hypo-eutectic steel, metallography....18, 90

I

PAGE
Inorganic compounds, table of molecular weights, etc., of.....118-121
Iron and steel, experiments on.....8, 10-16, 18, 27-30, 69-90
Iron, calorific power of..... 36
Iron, desulphurization of, by manganese..... 69
Isotherms 43

J

Junction, cold, of Le Chatelier pyrometer..... 5

L

Lampblack, lining crucibles with..... 115
Lead, desilverization of..... 24
Lead silicates, the fusibility of.....59, 60
Le Chatelier optical pyrometer..... 40
Le Chatelier thermo-electric pyrometer, calibration of....3, 5-8, 9, 38, 39
Le Chatelier thermo-electric pyrometer, cold junction of..... 5
Le Chatelier thermo-electric pyrometer, correction in reading..... 5
Le Chatelier thermo-electric pyrometer, precautions in using..... 5, 6
Le Chatelier thermo-electric pyrometer, principle of..... 3
Le Chatelier thermo-electric pyrometer, references on..... 9
Le Chatelier thermo-electric pyrometer, short-circuiting..... 6
Le Chatelier's experiment on alloy diffusion..... 107
Levol's alloy, composition of..... 18
Lime silicates, melting points of..... 126
Lime-alumina silicates, melting point determinations of.....55, 57
Lime-alumina silicates, melting points of..... 126
Lime, its influence on melting point of lead silicates..... 60
Lime linings, preparation of..... 116
Lime-magnesia silicates, melting point determinations of.....53-55, 56
Lime-magnesia silicates, melting points of..... 126
Lining crucibles, directions for..... 115
Linings for crucibles, basic and neutral..... 116
Liquid asphalt for binding material, use of..... 116

M

Magnesia, corrosion of, by silicates.....62-69
Magnesia crucibles..... 117
Magnesia-lime silicates, melting point determinations of.....53-55, 56
Magnesia-lime silicates, melting points of..... 126
Magnesia linings, preparation of..... 116

PAGE
Magnetic properties of steel, effect of temperature on..... 12
Magnetic properties of steel, their relation to hardening power..... 12
Mahler's Berthelot calorimeter.....31-36
Manganese, desulphurization of iron by..... 69
Manganese steel, effect of heat-treatment on..... 73
Martensite 122, 123
Martensite in blister steel, distribution of..... 89
Melting point of aluminium, determination of..... 9
Melting point of copper, determination of..... 38
Melting point of platinum, determination of..... 38
Melting point of zinc, determination of..... 7
Melting points of silicates, determination of:
General directions.....46-49
Lead silicates.....59, 60
Lime-alumina silicates..... 55
Lime-magnesia silicates.....53-55, 56
Melting points of silicates, table of..... 126
Melting points, table of.....124, 125
Mesuré and Nouel optical pyrometer..... 40
Metallography: blister steel.....88, 89
Metallography: copper-silver alloys..... 17
Metallography: directions for polishing..... 17
Metallography: etching (hypo-eutectic steel), directions for..... 18
Metallography: examination of hypo-eutectic steel.....18, 90
Metallography: preparation of sections.....17, 18
Metallography, references on..... 19
Metallography, synoptical table for.....122, 123
Metcalf's experiment on fracture..... 12
Metcalf's experiment on fracture elaborated..... 90
Microstructural changes in steel during refining.....80-82
Microstructure (see Metallography).
Mixtures, method of, for temperature determinations..... 30
Molecular weights, etc., table of.....118-121
Muffle arrangement for silicate melting point determinations.....47, 48
Muffle, special, for silicate melting point determinations..... 50
Muntz metal, composition of..... 92
Muntz metal, fracture of..... 92

N

Neutral crucible linings, preparation of..... 116
Nitric acid, correction for formation of..... 35
Nitric acid, heat of formation of..... 35
Nitric acid, quantitative determination of..... 35
Non-ferrous metals, experiments on.....91-114

O

PAGE
Ohm's law 105
Oil, heavy lubricating, its use as a binding material 46
Optical pyrometers 40
Overheated steel, refining of 14
Overstrain, growth of effects of 78
Overstrain, removal of effects of 29
Oxide (tempering) temperatures 124
Oxide tints accompanying tempering 27
Oxides, molecular weights, percentage compositions, etc., of 118, 119
Oxygen and sulphur, their relative affinity for copper 93

P

Parke's process for desilverizing lead 24
Pearlite 122, 123
Percentage compositions, etc., of various inorganic compounds, table of 118-121
Phosphates, molecular weights, percentage compositions, etc., of 120, 121
Platinum, melting point determination of 38
Polishing sections for microscopic examination, directions for 17
Process, chlorination, for gold 109
Process, copper selecting 91
Process, cyanide, for gold 108
Process, Elkington, copper electrolysis 104-107
Process, Parke's, for desilverizing lead 24
Process, Ziervogel, for argentiferous copper compounds 98-104
Purity of copper, influence of E. M. F. on 107
Purity of copper, spelter test for 92
Pyramids, preparation of silicate 46, 47
Pyramids, Seger 53
Pyrites, best percentage of, for chloridizing roasting 96
Pyrites, its effect in chlorination 113
Pyrometer, Le Chatelier thermo-electric (see Le Chatelier).
Pyrometer, Siemens' water 30, 31
Pyrometers, optical 40
Pyrometry and calorimetry, experiments on 30-46

R

Rail steel, finishing temperature for 88
Reaction of chloridizing roasting of copper ore 94
Reaction of cyanide process 108
Reaction, spangle 103
Reactions of Ziervogel process 100, 101

PAGE
Reduction of bars during rolling..... 86
References on alloy diffusion experiment..... 107
References on chloridizing roasting of copper ore..... 94
References on copper selecting process..... 92
References on finishing temperature..... 88
References on fracture of steel.....14, 78
References on Le Chatelier thermo-electric pyrometer..... 9
References on Mahler's Berthelot calorimeter..... 31
References on manganese steel..... 73
References on melting points of silicates..... 56
References on microscopic metallography..... 19
References on optical pyrometers..... 40
References on Parke's process..... 25
References on Seger pyramids..... 53
References on Siemens' water pyrometer..... 30
References on spelter test for purity of copper..... 93
References on Ziervogel process..... 98
Refining, microstructural changes in steel during.....80-82
Refining of overheated steel..... 14
Refining steel.....14, 80-82, 83-88
Refining steel, influence of T^{max} on thoroughness of.....83, 84
Refining steel, its effect on ductility..... 16
Refining steel, rationale of..... 80
Refractory materials, contraction of.....60-62
Refractory materials, corrosion of.....62-69
Refractory materials, slacking of basic..... 62
Rheostat for electrolysis..... 105
Roasting, chloridizing.....94-98
Roasting of copper sulphide ore, oxidizing.....19-24
Roberts-Austen's diagram, verification of..... 72
Rolling steel bars.....84-88

S

Sections for microscopic examination, preparation of.....17, 18
Seger pyramids..... 53
Selecting process, copper..... 91
Siemens' water pyrometer.....30, 31
Silicate pyramids, preparation of.....46, 47
Silicates, binding material for..... 46
Silicates, corrosion of refractory materials by.....62-69
Silicates, determination of melting points of (see Melting points)....46-60
Silicates, formation point of.....56-59
Silicates, molecular weights, percentage compositions, etc., of.....118-121
Silicates, relation of formation and melting points of.....56-59
Silicates, special muffle for determining melting points of..... 50
Silicates, table of melting points of..... 126

PAGE
Silver and gold, chlorination process for..... 109
Silver and gold, cyanide process for..... 108
Silver, sulphatization of.....102, 103, 104
Silver sulphide, roasting products of..... 102
Slacking of basic refractory materials..... 62
Slags, melting points, etc., of (see Silicates).
Slags, molecular weights, percentage compositions, etc., of.....118-121
Sorbite 122, 123
Spangle reaction..... 103
Spelter test for purity of copper..... 92
Stead's experiment on alloy diffusion..... 107
Steel and iron, experiments on.....8, 10-16, 18, 27-30, 69-90
Steel, burning hypo-eutectic.....82, 83
Steel, critical points of.....8, 70, 71, 72, 78, 85, 124, 125
Steel, finishing temperature for rail..... 88
Steel, finishing temperature for structural..... 88
Steel, fracture of.....12, 13, 16, 75-78, 90
Steel, manganese..... 73
Steel, refining of.....14, 80-82, 83-88
Steel, tool, Brinell's tests for.....75-78
Sulphatizing action of cupric sulphate.....102, 104
Sulphatizing action of ferrous sulphate.....101, 103
Sulphatizing action of ferrous sulphide..... 102
Sulphides, molecular weights, percentage compositions, etc., of....118, 119
Sulphur and oxygen, their relative affinity for copper..... 93
Sulphur, its influence in chloridizing roasting..... 98
Sulphuric acid, quantitative determination of..... 36
Supplies needed for copper roasting.....23, 24
Supplies needed for Parke's process..... 25
Synoptical table for metallography.....122, 123

T

Table for metallography, synoptical.....122, 123
Table of atomic weights..... 117
Table of boiling points.....124, 125
Table of comparison of thermometric scales..... 127
Table of important temperatures.....124, 125
Table of melting points.....124, 125
Table of melting points of silicates..... 126
Table of molecular weights, etc., of divers inorganic compounds....118-121
Table of percentage compositions of divers inorganic compounds..118-121
Table of special apparatus needed..... 128
Temper colors..... 124
Temperature determinations (see Pyrometry).
Temperature, finishing.....84-88
Temperature for chloridizing roasting..... 96

PAGE
Temperature, hardening, of steel....10, 12
Temperature, its effect on hardening power of steel....10-12, 27-29
Temperature, its effect on magnetic properties of steel.... 12
Temperature, its influence on refining....83, 84
Temperature limits for roasting copper sulphide ore....19-21
Temperature limits for Ziervogel process.... 100
Temperature uniformity determinations....41-44, 49
Temperatures, color names for....124, 125
Temperatures, formulæ for converting.... 127
Temperatures, table of important....124, 125
Tempering of steel....27-29
Thermal gradient determinations....41-44
Thermometric scales, table of comparison of.... 127
Tests, drop, directions for making.... 14
Tool steel, Brinell's tests for....75-78
Troostite....122, 123

U

Uniformity of temperature determinations....41-44, 49
Uniformity, relative, of gas and direct-fired furnaces.... 45

V

Vaseline for binding materials, use of.... 116

W

Weights, atomic, table of.... 117
Weights, molecular, etc., of divers inorganic compounds, table of...118-121

Z

Ziervogel process for argentiferous copper compounds....98-104

The Metallographist

A QUARTERLY PUBLICATION DEVOTED TO THE STUDY OF METALS, WITH SPECIAL REFERENCE TO THEIR PHYSICS AND MICROSTRUCTURE, THEIR INDUSTRIAL TREATMENT AND APPLICATIONS.

EDITED BY **ALBERT SAUVEUR**

PUBLISHED BY **THE BOSTON TESTING LABORATORIES**
446 Tremont Street, BOSTON, MASS.

The first volume of **The Metallographist** was published in 1898 and one volume has been issued in every subsequent year.

These volumes constitute an **exhaustive,** profusely illustrated treatise on metallography, indispensable to the student, a work of great value to the progressive metal manufacturer, worker or consumer, of much interest to the chemist, physicist and scientist in general.

Price: Bound in Cloth, $4.00 per volume.
Postage prepaid.

Special price to subscribers:

any volume	$3.75
any 2 volumes	$7.00
any 3 volumes or more, per vol. .	$3.25
Subscription per annum	$3.00

N. B.—Subscribers to The Metallographist are entitled to a discount of 10 per cent on all publications of the Boston Testing Laboratories, when ordered directly from the publishers.